How to cite: *Angew. Chem. Int. Ed.* **2023,** *62,* e202301486
International Edition: doi.org/10.1002/anie.202301486
German Edition: doi.org/10.1002/ange.202301486

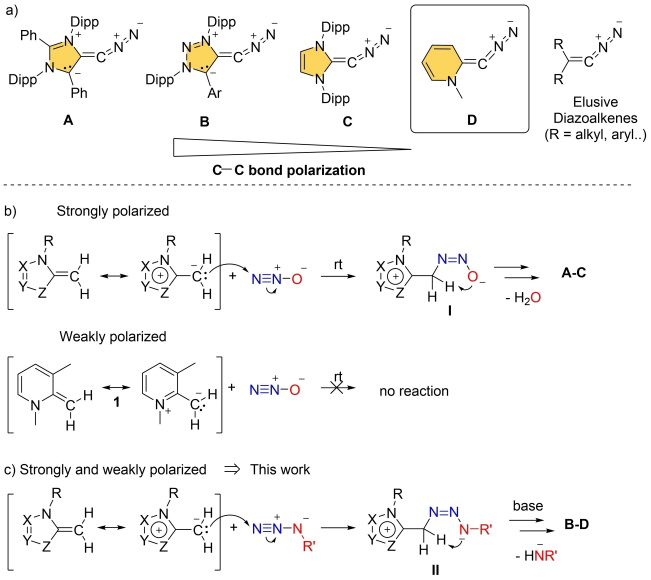
Diazo Compounds

Room-Temperature-Stable Diazoalkenes by Diazo Transfer from Azides: Pyridine-Derived Diazoalkenes

*Justus Reitz, Patrick W. Antoni, Julian J. Holstein, and Max M. Hansmann**

Dedicated to Guy Bertrand on the occasion of his 70th birthday

Abstract: Recently, stable diazoalkenes have received significant attention as a new substance class in organic chemistry. While their previous synthetic access was exclusively limited to the activation of nitrous oxide, we here establish a much more general synthetic approach utilizing a Regitz-type diazo transfer with azides. Importantly, this approach is also applicable to weakly polarized olefins such as 2-pyridine olefins. The new pyridine diazoalkenes are not accessible by the activation of nitrous oxide, allowing for a considerable extension of the scope of this only recently accessed functional group. The new diazoalkene class has properties distinct from the previously reported classes, such as photochemically triggered loss of dinitrogen affording cumulenes and not C–H insertion products. Pyridine-derived diazoalkenes represent the so far least polarized stable diazoalkene class reported.



Scheme 1. a) Known room temperature stable diazoalkene classes **A**–**C**. Dipp: 2,6-diisopropylphenyl; b) Activation of N_2O with only strong nucleophiles leads to diazoalkenes and c) proposed N_2 transfer with azides to access novel diazoalkenes.

Diazoalkenes ($R_2C{=}C{=}N_2$) are important intermediates in organic synthesis postulated for instance in the Seyferth–Gilbert homologation.[1] Since diazoalkenes are typically rapidly liberating dinitrogen gas to form fleeting vinylidenes,[2] there is few spectroscopic data available mostly derived from matrix isolation[3] or computations.[4] In 2021, we described the first room temperature stable diazoalkene **A** (Scheme 1), derived from the reaction of a mesoionic N-heterocyclic olefin (mNHO)[5] with nitrous oxide.[6] Shortly after, two additional stable diazoalkene classes based on a 1,2,3-triazole backbone (**B**),[7] as well as a 2-imidazole backbone (**C**)[8] were reported by us and the Severin group. The new diazoalkenes show unique chemistry, such as N_2/CO exchange,[7,9] as precursors to access triplet vinylidenes,[10] or to generate metal-vinylidene complexes.[11] Importantly, all the reported room-temperature-stable diazoalkenes were synthesized by the activation of nitrous oxide, which is intrinsically a challenging molecule to activate.[12] The mechanism is proposed to be initiated by addition of the strongly polarized carbon nucleophile onto N_2O to give zwitterionic alkane diazotate **I**,[13] which upon dehydration forms the desired diazoalkene (Scheme 1b; top). The utilization of nitrous oxide as a diazo transfer reagent, even though being highly atom-economic, has limitations: the outcome of the N_2O activation seems to be dependent on the exact reaction conditions,[14] and water has to be scavenged. Most importantly, the approach is restricted to strongly polarized carbon nucleophiles which are capable of activating nitrous oxide.

The synthesis and detection of diazoalkenes has been unsuccessfully targeted by several groups including Gilbert et al.[1,15] We could show that strong C–C bond polarization leads to stable diazoalkenes,[6] however, a fundamental question remains, as how much polarization is required to obtain room-temperature-stable diazoalkenes. Since pyridine derivatives are ubiquitous and electron deficient heterocycles, we were interested in such a structurally simple but yet unknown compound class **D** (Scheme 1). In compar-

[*] J. Reitz, P. W. Antoni, Dr. J. J. Holstein, Jun.-Prof. Dr. M. M. Hansmann
Fakultät für Chemie und Chemische Biologie, Technische Universität Dortmund
Otto-Hahn-Str.6, 44227 Dortmund (Germany)
E-mail: max.hansmann@tu-dortmund.de

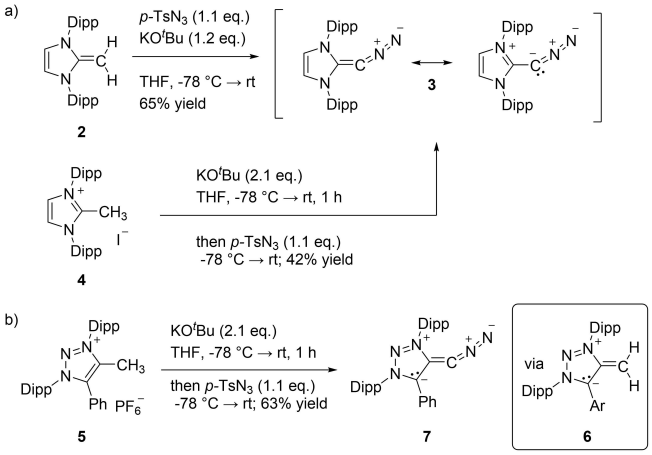
ison to the five membered rings (**A**–**C**), pyridine should be significantly more electron deficient, thus leading to the least polarized C=C bond in this series and consequently towards the direction of non-polarized elusive diazoalkenes (Scheme 1). We were curious if such a diazoalkene target could be stable at room-temperature and how to synthetically access it. Following the previous strategy, we attempted its synthesis by reacting the known 2-pyridine olefin **1**,[16,17] with N_2O but observed no reaction at room temperature (Scheme 1b). Forcing reaction conditions (90 °C for 5 days) lead to the scission of the C=C bond (see Supporting Information).

The lack of reactivity is most likely due to the lower nucleophilicity of 2-pyridine olefins compared to regular NHOs[18] and mNHOs[5] hampering the initial N_2O activation step. This is supported by the low field shifted ^{13}C NMR data for the exocyclic $=CH_2$ moiety of the pyridine olefin ($\delta \approx 67$–73 ppm),[16] compared to regular NHOs ($\delta \approx 40$–50 ppm), or mNHOs ($\delta \approx 40$–45 ppm). To solve the intrinsic problem of N_2O activation, we were curious if dinitrogen transfer would be feasible utilizing azides. One of the well-established ways to access diazoalkanes is the diazo transfer from azides, known as Regitz diazo transfer.[19] Already in 1953, Doering and DePuy reported that diazocyclopentadiene can be accessed by the reaction of *p*-tosyl azide with cyclopentadienyl lithium.[20] We hypothesized that C-nucleophiles such as ylidic olefins should add to azides leading to intermediate **II** (Scheme 1c), which was shown with 5-unprotected mNHOs but resulted in the generation of fused pyrazole heterocycles.[21] We speculated that additional base should facilitate deprotonation of **II** and N–N bond cleavage to lead to diazoalkenes and a sulfonamide salt byproduct. Importantly, azides should be significantly more electrophilic than nitrous oxide, increasing the applicability to weak nucleophiles and therefore to entirely new diazoalkene classes.

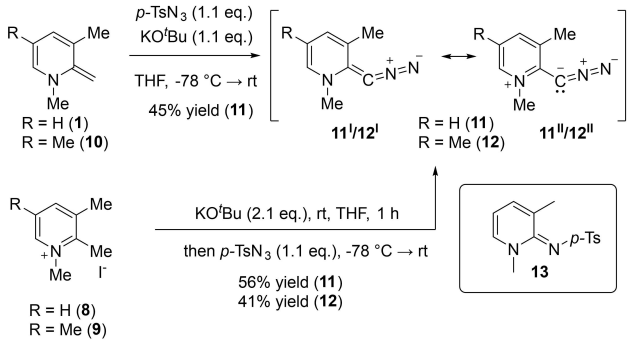
To test this hypothesis, we treated NHO **2**[22] with *p*-tosyl azide in the presence of KO^{t}Bu (Scheme 2a). We were delighted to isolate the known diazoalkene **3**[8] in 65 % yield. The deprotonated sulfonamide salt byproduct could easily be separated by precipitation. Note, **3** was previously only accessible by N_2O activation in DMF over 2 days reaction time,[8] and in CH_3CN afforded a dimerization product.[14] Since NHO **2** is synthesized from methylated imidazolium salt **4**, we attempted the one-pot synthesis starting from **4** with 2.1 equivalents KO^{t}Bu. Indeed, **3** could be isolated in 42 % yield, which represents a very convenient and rapid synthesis without the isolation of the air-sensitive olefin. We next selected triazolium salt **5** as precursor, which under the one-pot reaction conditions leads via mNHO **6**[5] to diazoalkene **7**[7] in 63 % yield (Scheme 2b). Other azides such as 2,4,6-triisopropylphenylsulfonyl azide, mesityl azide or *p*-CF_3-aryl azide also afforded diazoalkene **7**, but the clean isolation of the product proved more challenging.

Next, we applied this strategy to pyridine derived olefin **1**, which proved unreactive towards N_2O at room temperature (Scheme 1b). Interestingly, utilizing the azide strategy, olefin **1** and *p*-tosyl azide afforded a yellow solid in 45 % yield with a characteristic and intense IR band at 1971 cm^{-1}, which proved to be the unknown diazoalkene **11** (Scheme 3). **11** is stable under inert conditions at room temperature in solution or in the solid-state at least for more than 24 h. ^1H NMR data of the heterocyclic core show high field shifted signals at $\delta = 5.95$, 5.88 and 5.27 ppm indicating a localized non-aromatic structure **11^{I}** over a pyridinium **11^{II}** (Scheme 3), revealing a significant contribution of a genuine $R_2C{=}C{=}N_2$ diazoalkene. Besides **11**, we also observed the formation of ca. 10–20 % imine byproduct **13** in which formally a CH_2-fragment is lost (for X-ray verification and a mechanistic rationale, see the Supporting Information). The one-pot transformation starting from the pyridinium salt **8** also afforded the desired diazoalkene **11** in a single step. Additionally, we could perform the reaction with 1,2,3,5-collidinium salt **9** to give diazoalkene **12** (Scheme 3).

We were able to obtain single crystals suitable for X-ray diffraction of **12** (Figure 1).[23] Strikingly, the diazoalkene features a bent CCN unit (≈126°) and short C1–N1 (1.273 (4) Å/1.287(4) Å) and long N1–N2 (1.171(4) Å/1.163(4) Å) bond lengths (two molecules in the asymmetric unit). This trend agrees with the recent data for the structural parameters of diazoalkenes of type **A**–**C** (Scheme 1a). The CN_2 fragment is in plane with the heterocycle



Scheme 2. Synthesis of reported diazoalkenes **3** and **7** by diazo transfer with *p*-tosyl azide.



Scheme 3. Synthesis of pyridine derived diazoalkenes starting from the olefin (top) and from the salt precursor (bottom).

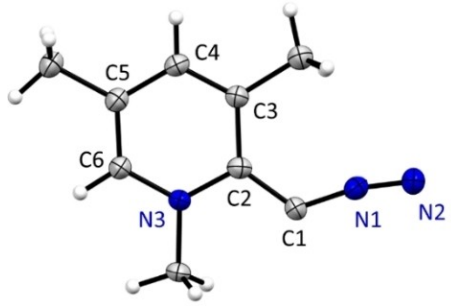
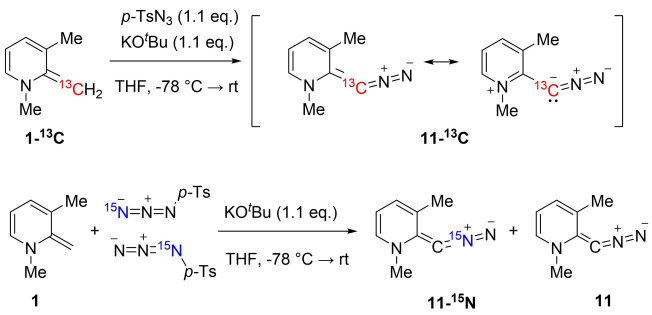


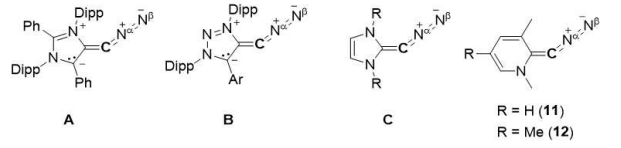
Figure 1. X-ray solid state structure of **12**. Thermal ellipsoids are shown with 50% probability. Selected bond lengths and angles in [Å] and [°] (two independent molecules in the asymmetric unit): C1–C2 1.402(4)/1.398(4); C1–N1 1.273(4)/1.287(4); N1–N2 1.171(4)/1.163(4); C2–C3 1.425(5)/1.437(4); C3–C4 1.379(5)/1.372(4); C4–C5 1.417(4)/1.402(5); C5–C6 1.341(5)/1.368(4); C6–N3 1.377(4)/1.371(4); C2–C1–N1 125.9(3)/125.5(3); N3–C2–C1–N1 178.9(3)/178.1(3).

(∢N3–C2–C1–N1: 179°) and oriented *trans* supporting a double bond contribution between C1 and C2, with a bond length of 1.402(4) Å/1.398(4) Å positioned between a single and double bond. In the heterocycle there is a pronounced



Scheme 4. ^{13}C and ^{15}N labeling studies.

Table 1: Comparison of selected spectral data of reported diazoalkenes (**A**–**C**) and pyridine diazoalkenes **11**/**12**.



Compound	A[9]	B[10]	C[11] (R = Ar or Me)	11/12
δ ^{13}C (CN_2) [ppm]	31.1	33.7-35.2	33.3-35.1 (N-Ar) 28.6 (N-Me)	66.0 (**11**) 63.2 (**12**)
^{13}C-^{13}C [Hz]	66	60	n. d.	75 (**11**)
δ ^{15}N [ppm] N^{α}/N^{β}	256.7/ 282.4	255.0/ 288.7	n. d.	256.6/ 279.6
IR [cm^{-1}]	1944	1953-1956	1961-1984 (N-Ar) 1954 (N-Me)	1971/1955 (**11**) 1951 (**12**)

C–C bond alternation (C2–C3≈1.43 Å; C3–C4≈1.37 Å) supporting the ^{1}H NMR data of localized double bonds.

In order to get insight into the electronic properties and the mechanism of the transformation, we performed ^{13}C and ^{15}N labeling studies (Scheme 4). Non-labeled **11** features a very weak ^{13}C signal (CNN) positioned at $\delta \approx 66$ ppm which increases its intensity upon cooling to −80 °C in d_8-THF (see Figure S86). Importantly, the intense ^{13}C signal (65.7 ppm) of **11-^{13}C** is downfield shifted compared to diazoalkenes **A**–**C**, which all appear in a narrow range of 30–35 ppm (Table 1) clearly highlighting the significant difference to the reported diazoalkenes. The ^{13}C shift indicates a trend towards a typical ^{13}C alkene range and supports a pronounced C=C double bond. Additionally, the labeled compound allows to derive the ^{13}C–^{13}C coupling constant of 75 Hz, which is the largest reported for diazoalkenes (Table 1). It is only slightly smaller than in the olefin (80 Hz) and significantly larger than in the C–C single bond of **8-^{13}C** (47 Hz, see Supporting Information), supporting a C=C double bond and a polarization towards a genuine diazoalkene. Next, we performed the reaction of **1** with ^{15}N labeled *p*-tosyl azide (50 % ^{15}N at inner and outer N-atoms, see Supporting Information). In relation to the natural abundance ^{15}N measurement only the inner N-atom [$\delta(^{15}N)$ = 256.6 ppm] is labeled (see Supporting Information). Therefore, the diazo transfer should mechanistically occur through attack on the outer (or inner) N-atom and not by attack on the middle N-atom of the azide. The ^{15}N shifts as well as the IR bands are comparable to systems **A**–**C** (IR range of ca. 1940–1980 cm^{-1}; Table 1).

The DFT optimized structural parameters agree well with the obtained solid-state structure (Figure 2a). The calculated natural charge of −0.22 e^- for the carbon atom (CNN) is the smallest compared to all model systems **A**–**C** (see Figure S113). Additionally, the Wiberg bond index for the C–CN_2 bond is the highest following the calculated trend 1.35 (**A**), 1.37 (**B**), 1.39 (**C**) and 1.43 (**11**). Calculations of the *E*/*Z* isomers show a favor for the *E* isomer by 2.0 kcal mol^{-1} in agreement with the solid-state structure (Figure 2b). Presumably, both *E*/*Z* isomers exist in solution, though low

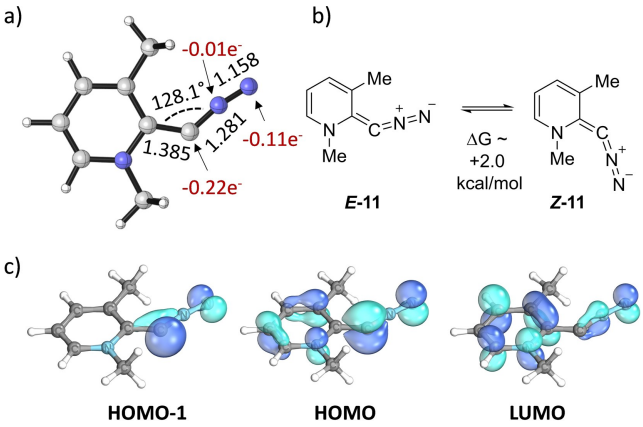
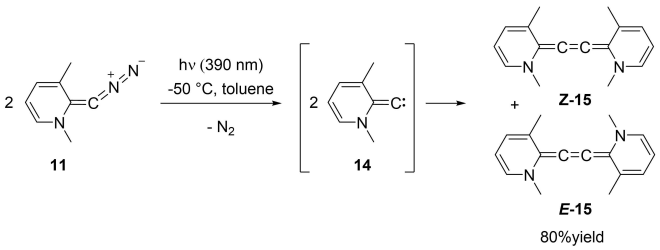


Figure 2. a) Calculated (BP86/def2-TZVPP) bond parameter; bond lengths in [Å] and natural charges in red; b) calculated energy difference between *trans*/*cis*-**11**; c) frontier molecular orbitals (isovalues at 0.6).

temperature NMR spectroscopy does not allow their separate detection. While HOMO-1 is centered on the CN_2 moiety, HOMO and LUMO are distributed over the entire π-system (Figure 2c). The frontier molecular orbitals, π-type character at C for HOMO and σ-type for HOMO-1, appear similar to the frontier molecular orbitals of the previously reported diazoalkene classes.[6,8a]

Due to the low molecular weight ($M \approx 147\ g\,mol^{-1}$) and high N/C ratio we performed thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of **11** and **12** (see Supporting Information). The TGA measurement of **11** shows a sharp mass loss of 77 % around 90 °C (Figure S103). DSC measurements of **11** in high-pressure crucibles confirm onset temperatures of ca. 88–95 °C. In the DSC measurements for both **11** and **12** two scenarios were encountered: either an initial decomposition followed by a second decomposition with moderate enthalpies of decomposition ($\Delta H_D \approx -425$–$505\ J\,g^{-1}$ and -216–$425\ J\,g^{-1}$), or an instant, highly energetic decomposition with $\Delta H_D \approx -1600\ J\,g^{-1}$ at ca. 90 °C (see Supporting Information). The later DSC profiles indicate new energetic materials exceeding by far ΔH_D values of all reported diazoalkenes (**A** and **B** $\approx 300\ J\,g^{-1}$; see Supporting Information) and several common diazo compounds.[24] Based on the DSC results suitable precautions (explosion shield, only small quantities) should be taken handling these energetic compounds.

Finally, we investigated the reactivity of the new diazoalkene class upon photochemical excitation. Irradiation of a solution of **11** with a 390 nm LED cleanly affords a ca. 1:1 mixture of *E*/*Z* dimerization products **15** (Scheme 5). This reactivity is in stark contrast to the reactivity of all previously reported diazoalkenes (**A**–**C**; Figure 1) which yield a range of C–H activation products, but importantly not dimers.[6–8] **15** features a characteristic ^{13}C NMR shift of the central C-atom at $\delta = 133$ ppm, which we could unambiguously assign by ^{13}C labeling [in agreement with theoretical prediction (Figure S117) and X-ray data (Figure S90).[23] Interestingly, there is only a single report on a 4-substituted C4 pyridine cumulene, which is generated by KC_8 reduction and discussed as pyridylidene stabilized dicarbon (py-> C_2 <-py).[25] The mechanism of the generation of C4 cumulene should involve the dimerization of vinylidene **14**.[26] Note, dimerization of other vinylidene precursors to give cumulenes has been experimentally observed,[27] but there has been serious doubt that this process actually involves free vinylidenes but instead metalated, carbenoid species.[2a] Due to the metal free and clean photochemical access, carbenoids can be ruled out here which should give strong support for a vinylidene pathway.[26] Note, that the reactivity of the pyridine diazoalkene starts to resemble the chemistry of typical elusive diazoalkenes.[2] It is interesting to point out that if diazoalkene **C** (Figure 1) would hypothetically dimerize (NHC)C_2(NHC) would be generated which is a compound studied in great detail theoretically[28] and was proven very unstable and easily decomposing into tetrazafulvalene and other products hampering its isolation.[29] Clearly, the photochemistry of the new pyridine diazoalkene class highlights the very different electronic nature and unique reactivity.



Scheme 5. Irradiation of **11** leads to C4 cumulene **15**.

In summary, we have reported a novel strategy to access stable diazoalkenes by dinitrogen transfer from azides. The only previously known method involved the activation of nitrous oxide, which is only applicable to strongly polarized olefins. This new approach enables the use of weakly polarized olefins, providing access to several so far unknown diazoalkenes by employing standard laboratory chemicals. The here presented pyridine-derived diazoalkenes show distinct properties such as a ^{13}C NMR shift (CN_2) of ca. 66 ppm compared to 28–35 ppm for all other previous systems and push the limits of bond polarization towards a genuine (non-polarized) diazoalkene featuring both C=C and C=N double bonds ($R_2C{=}C{=}N_2$). Remarkably, photochemically triggered loss of dinitrogen affords a stable C4-cumulene, while all other previous diazoalkene classes afforded C–H insertion products. Consequently, the diazoalkene class should be a highly interesting vinylidene precursor for main group or transition metal chemistry, which is under current investigation. Considering the exceeding simplicity of the synthetic access—pyridine derivatives are ubiquitous and just two commercially available reagents are added in a single synthetic step—we believe that this strategy will have a large impact on the exploration of the field. We are currently exploring other heterocycles employing this method and investigating the reactivity of pyridine diazoalkenes.

Acknowledgements

Sebastian Henke is thanked for access to the TGA and DSC instruments, Wolf Hiller for the ^{15}N NMR experiments as well as support by the faculty of chemistry and chemical biology. Thomas Klapötke is acknowledged for helpful discussions. The NMR spectrometer was funded by the Deutsche Forschungsgemeinschaft (DFG project 452669591). Computational resources were provided by LIDO3, the high-performance computing facility at TU Dortmund (DFG project 271512359). We thank the ERC-StG "CC-CHARGED" (101077332) and are looking forward to deepen the understanding of new carbon compounds within this project. Open Access funding enabled and organized by Projekt DEAL.

Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the Supporting Information of this article.

Keywords: Diazoalkenes · Reactive Intermediates · Diazo Compounds · Vinylidenes · Nitrogen Heterocycles

[1] a) J. C. Gilbert, U. Weerasooriza, *J. Org. Chem.* **1982**, *47*, 1837–1845; b) W. Kirmse, *Angew. Chem. Int. Ed. Engl.* **1997**, *36*, 1164–1170.
[2] a) P. J. Stang, *Chem. Rev.* **1978**, *78*, 383–405; b) R. Knorr, *Chem. Rev.* **2004**, *104*, 3795–3849.
[3] a) J. C. Brahms, W. D. Dailey, *J. Am. Chem. Soc.* **1990**, *112*, 4046–4047; b) J. Breidung, H. Bürger, C. Kötting, R. Kopitzky, W. Sander, M. Senzlober, W. Thiel, H. Willner, *Angew. Chem. Int. Ed.* **1997**, *36*, 1983–1985.
[4] M. A. Murcko, S. K. Pollack, P. M. Lahti, *J. Am. Chem. Soc.* **1988**, *110*, 364–368.
[5] M. M. Hansmann, P. W. Antoni, H. Pesch, *Angew. Chem. Int. Ed.* **2020**, *59*, 5782–5787.
[6] P. W. Antoni, C. Golz, J. J. Holstein, D. A. Pantazis, M. M. Hansmann, *Nat. Chem.* **2021**, *13*, 587–593.
[7] P. W. Antoni, J. Reitz, M. M. Hansmann, *J. Am. Chem. Soc.* **2021**, *143*, 12878–12885.
[8] a) P. Varava, Z. Dong, R. Scopelliti, F. Fadaei-Tirani, K. Severin, *Nat. Chem.* **2021**, *13*, 1055–1060; b) K. Bott, *Chem. Ber.* **1987**, *120*, 1867–1871.
[9] W. Feuerstein, P. Varava, F. Fadaei-Tirani, R. Scopelliti, K. Severin, *Chem. Commun.* **2021**, *57*, 11509–11512.
[10] Y. Kutin, J. Reitz, P. W. Antoni, A. Savitsky, D. A. Pantazis, M. Kasanmscheff, M. M. Hansmann, *J. Am. Chem. Soc.* **2021**, *143*, 21410–21415.
[11] a) B. Kooij, Z. Dong, P. Varava, F. Fadaei-Tirani, R. Scopelliti, L. Piveteau, K. Severin, *Chem. Commun.* **2022**, *58*, 4204–4207; b) B. Kooij, P. Varava, F. Fadaei-Tirani, R. Scopelliti, D. A. Pantazis, G. P. Van Trieste III, D. C. Powers, K. Severin, *Angew. Chem. Int. Ed.* **2023**, *62*, e202214899.
[12] K. Severin, *Chem. Soc. Rev.* **2015**, *44*, 6375–6386.
[13] R. A. Moss, *Acc. Chem. Res.* **1974**, *7*, 421–427.
[14] L. Y. M. Eymann, P. Varava, A. M. Shved, B. F. E. Curchod, Y. Liu, O. M. Planes, A. Sienkiewicz, R. Scopelliti, F. F. Tirani, K. Severin, *J. Am. Chem. Soc.* **2019**, *141*, 17112–17116.
[15] G. F. Hofmann, Versuche zur Darstellung und zum Nachweis von Diazoalkylidenen und Alkylidencarbenen, PhD thesis, **1973**, Universität Göttingen.
[16] a) A. Fürstner, M. Alcarazo, R. Goddard, C. W. Lehmann, *Angew. Chem. Int. Ed.* **2008**, *47*, 3210–3214; b) X. Zhang, L. Wang, Q. Wang, F. Ge, X. Wuang, *ChemistrySelect* **2019**, *4*, 8655–8660.
[17] The olefin without the methyl group in C3 position turned out to be unstable at room-temperature, therefore we selected olefin **1** as the simplest system.
[18] a) M. M. D. Roy, E. Rivard, *Acc. Chem. Res.* **2017**, *50*, 2017–2025; b) S. Naumann, *Chem. Commun.* **2019**, *55*, 11658–11670; c) R. S. Ghadwal, *Acc. Chem. Res.* **2022**, *55*, 457–470.
[19] a) M. Regitz, *Justus Liebigs Ann. Chem.* **1964**, *676*, 101–109; b) S. Bräse, C. Gil, K. Knepper, *Angew. Chem. Int. Ed.* **2005**, *44*, 5188–5240.
[20] W. von E. Doering, C. H. DePuy, *J. Am. Chem. Soc.* **1953**, *75*, 5955–5957.
[21] Q. Liang, K. Hayashi, Y. Zeng, J. L. Jimenez-Santiago, D. Song, *Chem. Commun.* **2021**, *57*, 6137–6140.
[22] S. M. Al-Rafia, A. C. Malcolm, S. K. Liew, M. J. Ferguson, R. McDonald, E. Rivard, *Chem. Commun.* **2011**, *47*, 6987–6989.
[23] Deposition Numbers 2232539 (for **12**), 2232538 (for **13**) and 2245896 (for **15**) 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.
[24] S. P. Green, K. M. Wheelhouse, A. D. Payne, J. P. Hallett, P. W. Miller, J. A. Bull, *Org. Process Res. Dev.* **2020**, *24*, 67–84.
[25] D. Wu, Y. Li, R. Ganguly, R. Kinjo, *Chem. Commun.* **2014**, *50*, 12378–12381.
[26] Preliminary calculations indicate a singlet ground state for **13** in contrast to all other triplet vinylidenes derived from **A**–**C**, see ref. [10], which requires additional investigations.
[27] a) D. Y. Curtin, W. H. Richardson, *J. Am. Chem. Soc.* **1959**, *81*, 4719–4728; b) G. Köbrich, W. Drischel, *Angew. Chem.* **1965**, *77*, 95.
[28] S. M. N. V. T. Gorantla, S. Pan, K. C. Mondal, G. Frenking, *J. Phys. Chem. A* **2021**, *125*, 291–301.
[29] D. C. Georgiou, I. Mahmood, M. A. Haghighatbin, C. F. Hogan, J. L. Dutton, *Pure Appl. Chem.* **2017**, *89*, 791–800.

Manuscript received: January 30, 2023
Accepted manuscript online: March 3, 2023
Version of record online: April 4, 2023