factor of 50 000 for MNBDH). The instant tea was weighed and dissolved in bidistilled water (0.5 g in 10 mL water) and diluted in the same way.

All measurements were carried out using microplate readers.

Glucose oxidase (GOD) reaction (I): 50 μL of a solution of GOD (15 mg) in acetate buffer (10 mL; pH = 5.5; 0.01M) were added to 100 μL of a glucose solution. After mixing thoroughly, the solution was incubated for 15 min at 37 °C.

GOD reaction (II): as I, but 20 μL GOD solution and 40 μL glucose solution were used.

GOD reaction (III): as I, but the GOD solution was prepared with phosphate buffer (pH 5.8; 0.01M).

Glucose determination using POD and ABTS: 50 μL of a solution containing POD (0.5 mg) and ABTS (5.5 mg) in acetate buffer (10 mL; pH 5.5; 0.01M) were pipetted to the mixture of reaction I. After the sample had been mixed thoroughly and incubated for 10 min at room temperature, the absorbance of the samples was measured at 405, 649, and 732 nm.

Glucose determination using POD and pHPA: 50 μL of a solution containing POD (2.5 mg) and pHPA (7.6 mg) in ammonium buffer (10 mL; pH 9.5; 0.01M) were pipetted to the mixture of reaction II. After the sample had been mixed thoroughly and incubated for 15 min at room temperature, the fluorescence of the samples was measured at excitation and emission wavelengths of 320 and 405 nm, respectively.

Glucose determination using POD and MNBDH: 1 mg MNBDH was dissolved in 10 mL acetonitrile. Of this solution, 1.4 mL are added to a solution containing POD (2.5 mg) in phosphate buffer (10 mL; pH 5.8; 0.01M). From this mixture, 40 μL were pipetted into the mixture of reaction III. After the sample had been mixed thoroughly and incubated for 10 min at room temperature, the fluorescence of the samples was measured at excitation and emission wavelengths of 470 and 545 nm, respectively.

Received: December 6, 1999 [Z 14358]

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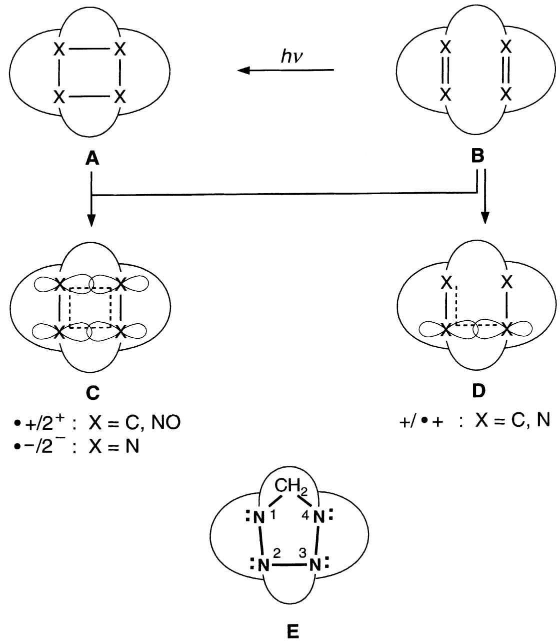
[10] ABTS is oxidized to an intensely green radical cation and shows UV/Vis absorption maxima at 405, 415, 649, 732, and 815 nm.
[11] The phenol derivative pHPA is dimerized in the peroxidase-catalyzed reaction to form the corresponding biphenol, which fluoresces in alkaline media (pH 9.5) with excitation and emission maxima of 323 and 403 nm, respectively.

σ-Homoconjugation in Cyclically Preoriented N4-(Radical) Cations—N···N Bond Lengths > 2 Å**

Kai Exner,* Birgit Großmann, Georg Gescheidt,
Jürgen Heinze, Manfred Keller, Thomas Bally,
Pavel Bednarek, and Horst Prinzbach*

(pp)σ-Conjugation, the electron delocalization between collinear p-orbitals, is a borderline case for the theory of chemical bonding.[1] It occurs efficiently only in compounds difficult to synthesize and with specific steric/stereoelectronic prerequisites. Making use of cage-fixed cyclobutanes **A** (X = C, (iso)pagodanes), of preoriented dienes **B** (X = C, (iso)-pagodadienes/(seco)dodecahedradienes), and bisdiazenes/bisdiazenetetroxides **B** (X = N, NO), the effective σ-homoconjugation or σ-bishomoaromaticity in 3C/3(2)e cations **D** (X = C),[2] in 4C(N)/3(2)e cations **C** (X = C, NO),[3, 4] and in 4N/5(6)e anions **C** (X = N)[5] have been observed. Here, we



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[*] Dr. K. Exner, Prof. Dr. H. Prinzbach, Dr. M. Keller
Institut für Organische Chemie und Biochemie der Universität Freiburg
Albertstrasse 21, 79104 Freiburg (Germany)
Fax: (+49) 761-203-5987
E-mail: horst.prinzbach@organik.chemie.uni-freiburg.de

Dr. B. Großmann, Prof. Dr. J. Heinze
Institut für Physikalische Chemie der Universität Freiburg
Albertstrasse 21, 79104 Freiburg (Germany)

Priv.-Doz. Dr. G. Gescheidt
Institut für Physikalische Chemie der Universität Basel
Klingelbergstrasse 80, 4056 Basel (Switzerland)

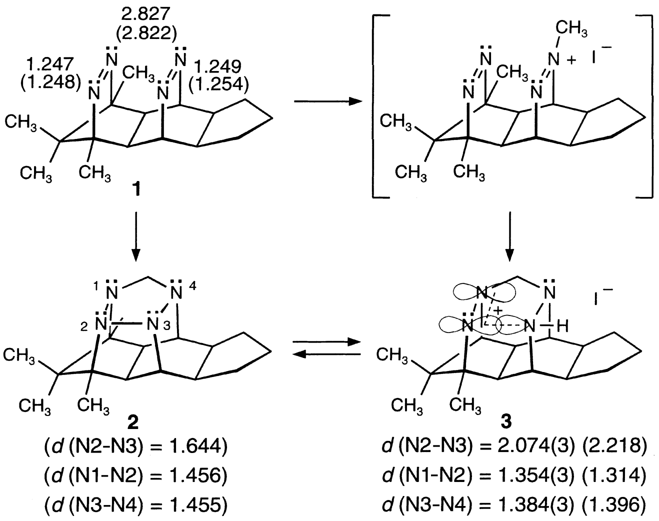
Prof. Dr. T. Bally, Dipl.-Chem. P. Bednarek
Institut für Physikalische Chemie, Universität Fribourg
Perolles, 1700 Fribourg (Switzerland)

[**] This project has been supported by the Deutsche Forschungsgemeinschaft, the Fonds der Chemischen Industrie and BASF AG. We thank Dipl.-Chem. V. Peron and B. Geiser for technical assistance.

Supporting information for this article is available on the WWW under http://www.wiley-vch.de/home/angewandte/ or from the author.

report on the σ-homoconjugation in tetraaza ions of type **D** (X = N), generated through the protonation and one-electron oxidation of type **E** tetrazolidines.[6] The latter—the first of their kind[7]—are all-*cis*-alkylated with four n-electron pairs ecliptically aligned and are accessible via the bisdiazenes **B**.[8]

The colorless tetrazolidine **2** (Figure 1; λ_{max}(relative ε) = 364 nm (13), 212 (100)[9]) was quantitatively produced through the treatment of the bisdiazene **1** with CH_3I ($t_{1/2}$ = 3.5 d) followed by treatment of the yellow, thermally quite stable salt **3** (decomp > 100 °C; $\lambda_{max}(\varepsilon)$ in CH_3CN = 350 nm (3070), in CH_3OH = 344 nm, in $CHCl_3$ = 335 nm) with aqueous K_2CO_3. Reprotonation of **2** leads back to **3**; according to calculations (B3LYP/6-31G* level) the protonation at N3 (numbering of **E**) is energetically favored.



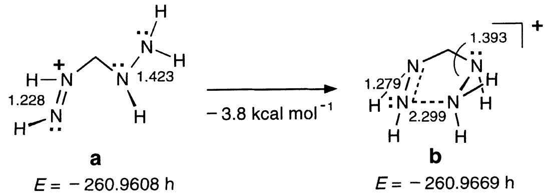
(*d* (N2-N3) = 1.644)
(*d* (N1-N2) = 1.456)
(*d* (N3-N4) = 1.455)

d (N2-N3) = 2.074(3) (2.218)
d (N1-N2) = 1.354(3) (1.314)
d (N3-N4) = 1.384(3) (1.396)

Figure 1. Selected experimental and (in parentheses) calculated (B3LYP/6-31G*) distances [Å] for **1**, **2**, and **3**.

The salt **3**, according to the X-ray structural analysis[10] and the B3LYP/6-31G* calculations,[11] features unusual bonding properties (Figure 1). As consequence of the strong electron repulsion in the tetraaza chain of **2**, the N2–N3 bond is considerably long (d = 1.644 Å), whereas the N1–N2 and N3–N4 bonds are of normal length. In the salt **3**, the N2–N3 bond is elongated by a further 0.430 Å, but the N1–N2 and N3–N4 bonds, in contrast, are shortened by 0.102 and 0.071 Å, respectively. In essence, **3** represents a diazenium ion (R_2N1^+=N2R), that is stabilized by σ-homoconjugation involving the n-electron pair on N3 of the hydrazine unit (N3–N4). According to an NBO analysis, the π* orbital of the N1=N2 double bond is occupied by 0.56 electrons and the N3 electron lone pair is the main donor, providing an interaction energy of 26.96 kcal mol^{-1}; the N4 lone pair remains largely localized.

Significant through-space interaction between N1 and N4, in the sense of cyclic delocalization (**C**), is excluded by a Bader analysis.[12] Only for the N2–N3 bond does a critical point exist, albeit with a small electron density of ρ = 0.0537 ea_0^{-3}. The corresponding Laplacian $\nabla^2(\rho) = +0.1488$ characterizes a donor–acceptor type bond;[13] the color of **3** results from a charge-transfer transition. The thermodynamic value of the N2 ··· N3 σ-homoconjugation over a 2 Å distance has been estimated for the model compound **4**: Of the two conformers **a** and **b** the σ-homoconjugated **b** is, by B3LYP/6-31G* calculations, more stable by 3.8 kcal mol^{-1}.

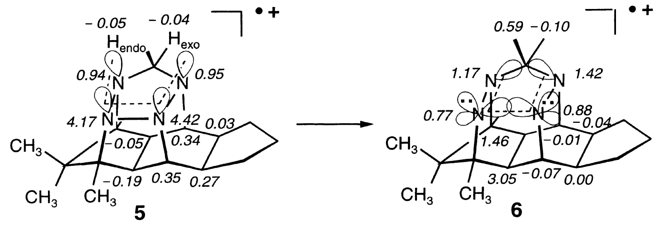


E = −260.9608 h

E = −260.9669 h

4

How does tetrazolidine **2** respond to the loss of an electron?[14] A B3LYP/6-31G* optimization starting from the structure of neutral **2** leads—in agreement with the nodal situation of the HOMO of **2**—with shortening of all N–N distances to the radical cation **5** (Figure 2).[15] The calculated isotropic hyperfine-coupling constants (hfcs) of **5** give dominant values for the four nitrogens of 4.42 and 4.17 mT for N2 and N3, and about 0.95 mT for both N1 and N4; the hfcs of the central protons, located in the nodal plane of the singly occupied molecular orbital (SOMO), are very small.



d (N2-N3) = 1.403
d (N1-N2) = 1.421
d (N3-N4) = 1.425

d (N2-N3) = 2.315
d (N1-N2) = 1.308
d (N3-N4) = 1.311

Figure 2. Calculated (B3LYP/6-31G*) hyperfine coupling constants [mT] (italics) and distances [Å] for the radical cations **5** and **6**.

In cyclovoltammetric experiments, **2** was reversibly oxidized and the oxidation potential was surprisingly high ($E_{1/2}$ = 0.56 V versus Ag/AgCl, 0.5 V s^{-1}, 281 K). The temperature dependence of the potential difference of ΔE_{EP} = 0.15 V (0.079 V at 298 K) indicates significant structural changes during the charge transfer process. Chemical oxidation of **2**, using diverse methods, leads to moss-green solutions (λ_{max} (CH_2Cl_2) = 751 nm), yet this radical does not belong to the $C_{17}H_{26}N_4^{\bullet+}$ hypersurface.[6, 16] In contrast, ionisation of **2** by γ-irradiation (^{60}Co) at 77 K (matrix CCl_3F/CF_2BrCF_2Br = 1:1) generated a deep-violet radical cation (λ_{max} = 560 nm). The EPR spectrum (Figure 3) proved incompatible (see Supporting Information) with the calculated spectra (B3LYP/6-31G*) of **5** and its retro[3+2]$^{\bullet+}$ cycloadduct, yet it was in good agreement with the calculation for the N2–N3-σ-homoconjugated species **6**. The formation of the latter formally occurs by loss of an electron from the HOMO-1 of **2** (which is nearly isoenergetic with the HOMO).[15]

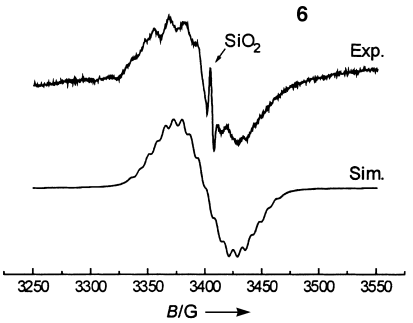


Figure 3. EPR spectra of **2** ionized at 77 K by γ-irradiation in a matrix (CCl_3F/CF_2BrCF_2Br 1/1; 111 K, modulation 0.3 mT) and simulation against B3LYP/6-31G* data.

The transformation **5**→**6**, calculated to be exothermic by 10.5 kcal mol^{-1}, proceeds with strong elongation (0.912 Å) of the N2–N3 bond to 2.315 Å and shortening (0.113 Å) of the N1–N2 and N3–N4 bonds. Unlike in **5**, the central hydrogen atoms are no longer in the nodal plane of the SOMO (Figure 4), causing drastic changes of the (calculated) ^1H coupling constants. This assignment is supported by the UV/Vis spectra calculated (Figure 5) for the radical cations **5** and **6** (and its retro[3+2]$^{\cdot+}$ cycloadduct); only for **6** is the experimental absorption well reproduced (λ_{max} (TD-B3LYP/6-31G*)[18] = 515 nm ($f = 0.0041$), 631 nm ($f = 0.0005$)). The two longest wavelength transitions are negative (shoulder at 630 nm) and positive (band at 560 nm) combinations of the SOMO→LUMO and SOMO-1→SOMO transitions. The transformation **5**→**6** is related to the "tight"→"extended" isomerization of the 4C/3e radical cations of the [1.1.1.1]pagodane, for which a barrier of $E_a = 2.2 \pm 0.3$ kcal mol^{-1} has been determined with the fast FDMR method.[3b]

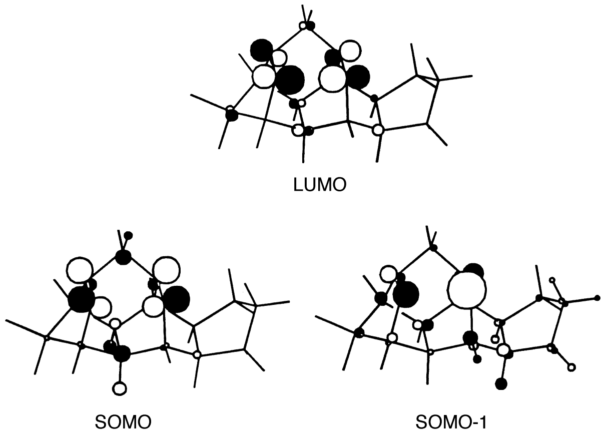


Figure 4. MOPlot[17] representation of the frontier orbitals of **6**.

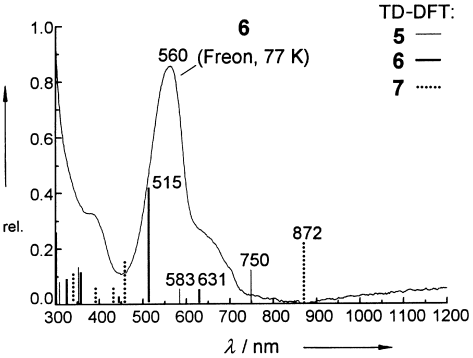


Figure 5. UV/Vis spectra of **2** ionized at 77 K by γ-irradiation (solid line) in a matrix (CCl_3F/CF_2BrCF_2Br 1/1). The results of the TD B3LYP/6-31G* calculations for **5** and **6** (retro[3+2]$^{\cdot+}$ cycloadduct = **7**) are shown as vertical lines.

Received: December 14, 1999 [Z14392]

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[9] **2**: (±)-(1*R**,2*S**,3*R**,4*S**,6*R**,11*S**,12*S**,16*R**)-4,5,5,6-Tetramethyl-7,8,10,17-tetraazaheptacyclo[8.6.1.0^{2,6}.0^{3,11}.0^{4,8}.0^{7,17}.0^{12,16}]heptadecane.

[10] Crystallization of **1** from $CHCl_3$ proceeds with spontaneous resolution and inclusion of a solvent molecule ($[\alpha]_D^{21} = 12.9$; $c = 1.4$ g L^{-1}). Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-137669. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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[15] The SOMO and SOMO-1 of the radical cation **5** corespond to the HOMO and HOMO-1 of neutral **2**. The HOMO of **2** is described as

the antibonding combination of the four nonbonding electron pairs at the nitrogen atoms.

[16] An additional wave is observed at about 0.01 V in the backsweep, which disappears at faster scan rates. The nature of the corresponding novel 4N/7e radical cation will be reported separately.K. Exner, B. Großmann, G. Gescheidt, J. Heinze, M. Keller, T. Bally, P. Bednarek, H. Prinzbach, unpublished results.

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Extremely Long, Discrete *meso*–*meso*-Coupled Porphyrin Arrays[**]

Naoki Aratani, Atsuhiro Osuka,* Yong Hee Kim, Dae Hong Jeong, and Dongho Kim

Considerable attention has been focused on the synthesis of monodisperse macromolecular rods of precise length and constitution in light of their potential application as molecular-scale electronics, optical devices, sensors, and for conversion of solar energy.[1–3] Discrete molecular rods of known structure are used to position two active centers at a known distance, and the resulting assemblies are of interest as potent electronic or photonic molecular wires. Recently, the length of linear, monodisperse, π-conjugated oligomers have reached the range of approximately 10 nm.[1, 4] Yet it still remains a great synthetic challenge to explore discrete, finite functional supramolecules with well-defined structures far beyond these achievements.

One of the most attractive building blocks for supramolecular rods are porphyrins, since they offer a variety of desirable features such as a rigid, planar geometry, high stability, an intense electronic absorption, a strong fluorescence emission, a small HOMO–LUMO energy gap, as well as flexible tunability of their optical and redox properties by appropriate metalation.[5] Recent efforts on the preparation of supramolecular porphyrin arrays have become increasingly focused on the realization of various molecular devices.[5, 6] However, these studies are often hampered by poor solubility, difficult separations, and demanding characterizations. Therefore, high solubilities, easy separations, and reliable characterizations of the arrays are of prime importance in devising a larger molecular system.

Recently we found that the Ag^I-promoted *meso*–*meso*-coupling reaction of Zn^{II} 5,15-diarylporphyrins has several advantages;[7, 8] 1) the regioselectivity of the *meso*–*meso* coupling is quite high, 2) the porphyrin arrays have essentially the same linear rodlike shape, 3) the porphyrin arrays are highly soluble, presumably because of orthogonal conformations arising from steric hindrance around the *meso*–*meso* linkage, 4) the separation of the coupling products is easy by recycling preparative GPC-HPLC chromatography as a result of large differences in molecular weight, and finally 5) the long coupling products still bear two free *meso* positions that are available for the next reaction.

Here we report the synthesis and characterization of *meso*–*meso*-coupled porphyrin oligomers up to 128-mer, which is, to the best of our knowledge, the longest (ca. 106 nm) monodisperse, rodlike molecule prepared so far. Previously we employed Zn^{II} 3,5-di-*tert*-butylphenylporphyrin as a building block, but we encountered a serious solubility problem at the stage of the porphyrin 8-mer. In order to circumvent the solubility problem, we employed the more soluble Zn^{II} 3,5-dioctyloxyphenylporphyrin **Z1** (here we denote the *meso*–*meso*-coupled Zn^{II} porphyrin arrays as ***Zn*** where ***n*** represents the number of porphyrins; Ar = 3,5-dioctyloxyphenyl). Chain elongation can be achieved quite simply by repeating the dimerization reactions from **Z1** to **Z2**, **Z2** to **Z4**, **Z4** to **Z8**, **Z8** to **Z16**, and **Z16** to **Z32**. The yields of the dimerization products were commonly 20–30 % together with the recovery of starting materials (55–60 %). Finally the coupling reaction of **Z32** afforded **Z64**, **Z96**, and **Z128** in yields of 25, 19, and 5 %, respectively, all in a discrete state. The longest porphyrin array **Z128** isolated from the reaction of **Z32** was identical to the product obtained from the dimerization of **Z64**. During these repeated preparations we also isolated **Z3**, **Z5**, **Z7**, **Z10**, **Z12**, **Z20**, **Z24**, **Z40**, and **Z48**. The use of pure $CHCl_3$ as the solvent and strictly controlling the reaction temperature to 30 °C were crucial to avoid polymerization of the Zn^{II} porphyrin, which was enhanced particularly in the presence of small amounts (0.5–3 %) of *N*,*N*-dimethylacetamide (DMA) or upon slight heating.[9] Also equally important was the use of recycling GPC-HPLC for product separation. Figure 1 shows GPC-HPLC chromatographs of the reactions of **Z16** and **Z32**. The coupling products were nicely separated by GPC-HPLC in both cases owing to the large differences in molecular weight of the products. The molecular weights of these porphyrin arrays were determined by matrix-assisted laser desorption ionization (MALDI-TOF) mass spectrometry (Table 1).[10]

Figure 2 shows the ^1H NMR spectra of **Z1**, **Z2**, **Z4**, **Z8**, **Z16**, **Z32**, **Z64**, and **Z128** taken in $CDCl_3$ at room temperature. To our surprise the longer arrays **Z64** and **Z128** display relatively well-resolved spectra similarly to those of the shorter arrays. The assignments were performed through comprehensive

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[*] Prof. A. Osuka, N. Aratani
Department of Chemistry
Graduate School of Science
Kyoto University
Sakyo-ku, Kyoto 606-8502 (Japan)
Fax: (+81) 75-753-3970
E-mail: osuka@kuchem.kyoto-u.ac.jp

Y. H. Kim, D. H. Jeong, Dr. D. Kim
National Creative Research Initiatives Center for Ultrafast Optical Characteristics Control
Korea Research Institute of Standards and Science (KRISS)
Tajeon 305-600 (Korea)

[**] This work was supported by Grant-in-Aids for Scientific Research (No. 11136221 and 11223205) from the Ministry of Education, Science, Sports, and Culture of Japan and by CREST (Core Research for Evolutional Science and Technology) from the Japan Science and Technology Corporation (JST). The work at KRISS was supported by the National Creative Research Initiatives of the Ministry of Science and Technology of Korea.

0570-0833/00/3908-1458 $ 17.50+.50/0