frames (0.3° in ω) and were corrected semiempirically for absorption (transmission 0.709–0.928). The structure was solved by direct methods and refined by full-matrix least-squares on F^2 values of all to give $wR2 = \{\Sigma[w(F_o^2 - F_o^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2} = 0.2202$ conventional $R = 0.0830$ for F values of 6700 reflections with $F_o^2 > 2\sigma(F_o^2)$, $S = 0.927$ for 1159 parameters. Residual electron density extremes were 0.267 and -0.221 e Å^{-3}. Highly disordered toluene solvent molecules were treated by the SQUEEZE procedure of PLATON (A. L. Spek, University of Utrecht, The Netherlands, **2000**), which indicated the correct total electron density for four molecules of toluene per molecule of **3**. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-166394 (**3**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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[13] Preliminary DFT calculations (B3LYP/6–31G*) for the simplified model system $[\{HC(CHNH)_2MgCH_3\}_2(\mu\text{-}\eta^1{:}\eta^1\text{-}C_3H_5)]^-$ were performed to obtain supporting evidence for sp^2 hybridization of the allyl CH$_2$ groups. The C–C bond lengths (1.403 Å) obtained clearly indicate an unsaturated bonding environment; in addition Mulliken charge assignments yield a positive charge to the central CH group, and negative charges to the neighboring CH$_2$ groups. The highest occupied molecular orbital (HOMO) can be largely attributed to p-type character orbitals located on the CH$_2$ carbon atoms, pointing in the direction of the Mg centers. Further details are provided in the Supporting Information.

An Unorthodox Conformation of [18]porphyrin-(1.1.1.1) Heteroanalogue—21,23-Ditelluraporphyrin with a Flipped Tellurophene Ring**

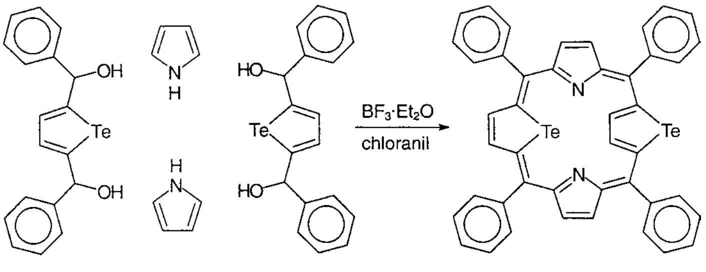
Ewa Pacholska, Lechosław Latos-Grażyński,* and Zbigniew Ciunik

Conformational flexibility of tetrapyrrole macrocycles is currently appreciated as an important factor in the fine-tuning of porphyrin and metalloporphyrin properties.[1] Thus, introduction of sterically demanding residues (including nitrogen atom protonation) in the porphyrin core or at the outer rim (alkylation, arylation), as well as coordination of metal ions, can result in a nonplanar porphyrin conformation. Core-modified porphyrins, formed by replacement of one or more nitrogen atoms by other heteroatom(s), also demonstrate severe nonplanar arrangements once the size and/or number of heteroatoms in the core increases, even though the basic framework of the porphyrin skeleton is preserved.[2] On the whole, [18]porphyrins-(1.1.1.1) and their heteroanalogues contain a similar framework of four essentially planar five-membered rings linked by methine carbon atoms with all the nitrogen atoms or other heteroatoms pointing toward the center of the macrocycle. Even the most severe distortion of a macrocycle preserves such an “in”-macrocycle orientation of the five-membered rings.[1]

An enlargement of the macrocycle by the addition of heterocyclic moiety(s) and/or methine carbon atoms introduces a novel structural motif. Thus a five-membered ring—axiomatic for porphyrins (heteroporphyrins)—can be oriented in such a way that one nitrogen or other heteroatom is located at the outer perimeter and two β-atoms are found in the core or just over the center. An inversion of this type has been detected for the following expanded porphyrins and expanded heteroporphyrins:[3] tetrathia[22]porphyrin-(2.2.2.2),[4] tetrathia[24]porphyrin-(2.2.2.2),[5] *meso*-substituted [22]pentaphyrin-(1.1.1.1.0) (sapphyrins)[6] and its heteroanalogues,[7, 8] [26]hexaphyrin-(1.1.1.1.1.1),[9] heteroanalogues of [26]hexaphyrin-(1.1.0.1.1.0) (rubyrin),[3, 10] and heteroanalogues of [30]heptaphyrins-(1.1.0.0.1.1.0) and [30]heptaphyrins-(1.1.0.1.0.1.0).[11] The lowest limit of the macrocycle size permitting the flipped geometry is exemplified by [18]annulene bridged by three sulfur atoms (which formally may be named trithia[18]porphyrin-(2.2.2)), but it behaves only as an array of isolated thiophene units which are totally out of plane in the solid state.[5] It was also postulated that a 180° rotation of the *N*-confused pyrrole ring of 2-aza-21-carbaporphyrin is necessary to produce the “fused” porphyrin.[12] The conformation of 2-aza-21-carbaporphyrin with the flipped pyrrole ring is energetically accessible as demonstrated by DFT calculations.[13]

Here we report on the synthesis and characterization of 5,10,15,20-tetraphenyl-21,23-ditelluraporphyrin (Te$_2$TPP, **2**) which is the first heteroanalogue of [18]porphyrin-(1.1.1.1) with the nonplanar macrocyclic conformation containing the flipped five-membered ring.

The 21,23-ditelluraporphyrin **2** was synthesized by condensation of pyrrole and 2,5-bis(phenylhydroxymethyl)tellurophene[14c] (**1** Scheme 1). This procedure follows the methodology previously used for the preparation of 5,10,15,20-



1 **2**

Scheme 1. Synthesis of Te$_2$TPP.

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[**] Financial support from the State Committee for Scientific Research KBN of Poland (Grant: 3T09A 155 15) and the Foundation for Polish Science is gratefully acknowledged.

tetraaryl-21,23-diheteroporphyrins.[14] The product was obtained in 11% yield after chromatographic workup.

Te_2TPP has been characterized by X-ray crystallography.[15, 16] The size of the tellurium atoms is instrumental in the remarkable distortion of macrocycle **2**. One of the tellurophene moieties (Te23) is essentially coplanar with two adjacent pyrrole rings while the second tellurophene ring is flipped and the Te21 atom is directed away from the center of the macrocycle. The geometry is apparent from the views of the macrocycle in Figure 1. The extent of distortion is reflected by a dihedral angle between the plane of *meso*-carbon atoms (C5-C10-C15-C20) and the tellurophene plane (Te21-C1-C2-C3-C4) of 123.0(2)°.

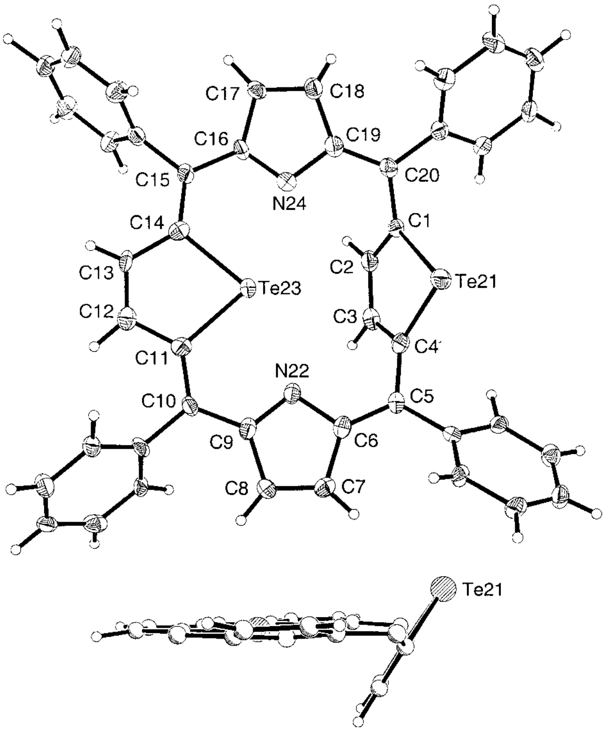


Figure 1. The crystal structure of Te_2TTP (top: perspective view; bottom: side view—the phenyl groups are omitted for clarity). The vibrational ellipsoids represent 50% probability. (Selected bond lengths [Å]: Te21-C1 2.087(8), Te21-C4 2.088(8), C1-C2 1.37(1), C2-C3 1.42(1), C3-C4 1.36(1), Te23-C11 2.107(8), Te23-C14 2.092(9), C11-C12 1.43(1), C12-C13 1.36(1), C13-C14 1.44(1)).

The electronic spectrum of **2** shows three major bands of comparable intensities at 348, 464, and 668 nm, but the distinct intense Soret-like band typical for aromatic heteroporphyrins is absent (Figure 2). The electronic spectrum of $[(Te_2TPP)H_2]^{2+}$ ($\mathbf{2}$-H_2^{2+}), shows the presence of a Soret-like band at 490 nm which is accompanied by a less-intense band at 767 nm. The change in the spectrum is explained by the more aromatic nature of the protonated form.

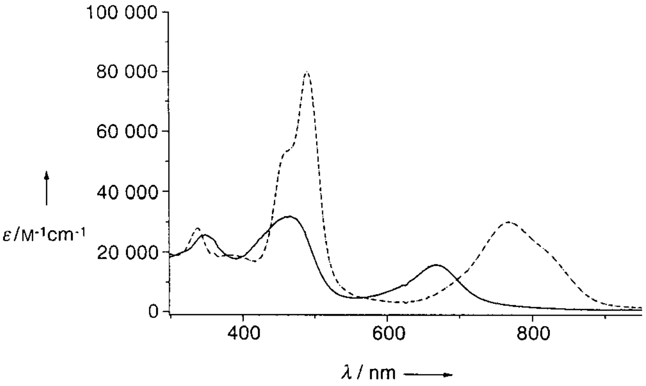


Figure 2. The electronic spectra of Te_2TTP (solid line) and $[(Te_2TTP)H_2]^{2+}$ (dashed line) in dichloromethane.

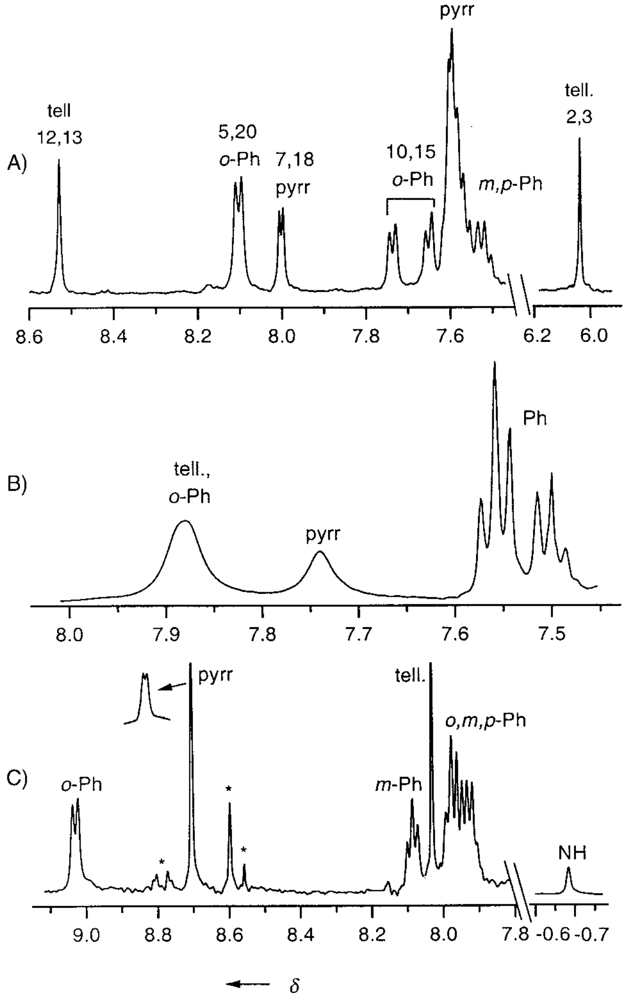


Figure 3. Partial ^1H NMR spectra in $CDCl_3$: A) Te_2TTP (210 K); B) Te_2TTP (298 K); C) $[(Te_2TTP)H_2]^{2+}$ (20 equiv of TFA; 210 K). Peak labels follow the systematic position numbering of the porphyrin ring or denote proton groups: *o*, *m*, *p* positions of the *meso*-phenyl groups.

The remarkable flipped structure of **2** detected in the solid state is preserved in solution, as determined by ^1H NMR studies. Unambiguous assignments of the resonances were obtained by a selective deuteration of pyrrole fragments to get $[D_4]Te_2$TPP, as well as by 2D ^1H NMR COSY and ROESY experiments. The ^1H NMR spectrum of **2** (210 K) exhibits one AB pattern centered at $\delta = 7.8$ ($^3J_{HH} = 4.5$ Hz, Figure 3A), which was readily assigned to the pyrrole ring. The definitive determination of the flipped conformation was provided by the diagnostic shifts of the tellurophene protons at $\delta = 8.53$ and 6.08, which were assigned to the regular and flipped rings, respectively. The aromatic ring current effect is small because of the severe distortion, as reflected here by the difference in shift between the inner and outer β-H tellurophene resonances ($\delta_{12} - \delta_2 = 2.45$).

Titration with trifluoroacetic acid (TFA) triggers a profound change in the geometry involving a rearrangement toward the "regular" (**R**) structure in which the Te21 and Te23 tellurophene atoms are simultaneously directed toward the center of the macrocycle (Scheme 2). A strong puckering of the macrocycle with large displacements of both tellurophene rings from the mean plane is expected because of the steric crowding. The "regular" structure of **2**-H_2^{2+} is evident in the ^{1}H NMR spectrum as two lines assigned to the tellurophene ($\delta = 8.03$) and pyrrole ($\delta = 8.71$, $^4J_{\text{H-NH}} = 1.5$ Hz) resonances (Figure 3C). The NH resonance located in the upfield region ($\delta = -0.7$) indicates the marked ring current effect. The puckering of the dication is confirmed by the very large differentiation in the *ortho* (ca. $\Delta\delta = 1$) and *meta* (ca. $\Delta\delta = 0.15$) resonances of the *meso*-phenyl groups.

The well-defined ^{1}H NMR signals of **2** measured in $CDCl_3$ at 210 K (Figure 3A) broaden, coalesce (Figure 3B, 298 K), and finally give one sharp peak for each position. These sharp peaks indicates there is a fast exchange process at 320 K (tellurophene: $\delta = 7.88$, pyrrole: $\delta = 7.74$). In fact, the "in" and "out" arrangements of the two inequivalent tellurophene rings of **2** are exchangeable even at 210 K, as proven by the EXSY correlation in the ROESY map. The Te_2TPP molecule interchanges between two energetically and structurally identical flipped forms **F**-1 and **F**-2 as shown in Scheme 2.

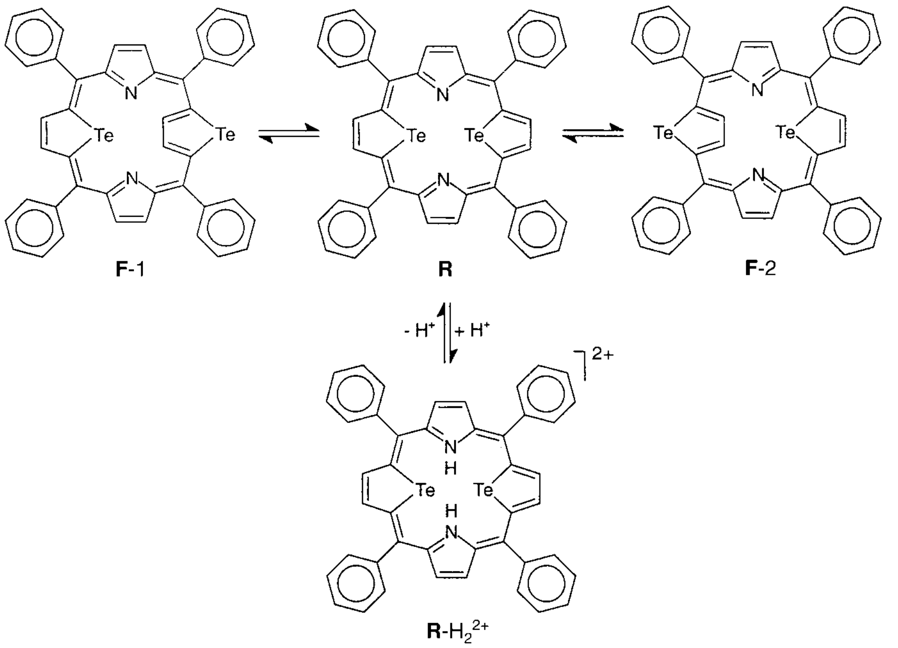
This mechanism implies a sequence of two steps which involves an approximate 180° rotation of the flipped (Te21) tellurophene ring of **F**-1 to form the regular conformation **R**, followed by the analogous flip of the Te23 tellurophene ring to form **F**-2. The Te_2TPP **R** form is not merely transient, but it makes a meaningful contribution to the thermodynamic equilibrium. Consequently, at 320 K the resonance positions differ from these expected for a two-site, equally populated exchange mechanism, namely, β-H tellurophene at $\delta = 7.30$ and β-H pyrrole at $\delta = 7.78$.

Attempts to broaden the basis of heteroporphyrin chemistry has stimulated interest in 21,23-ditelluraporphyrin, which has heretofore escaped all efforts of synthesis.[14c, 17] 21,23-Ditelluraporphyrin provides the first example of the inverted structure of the 18-π electron porphyrin-like frame with four five-membered rings linked by four methine carbon atoms.

Experimental Section

2: Pyrrole (65 mg, 0.97 mmol), **1** (0.37 g, 0.94 mmol), and freshly distilled CH_2Cl_2 (400 mL) were placed in a 1000-mL flask with a reflux condenser. Nitrogen was bubbled through the solution for 20 min, then $BF_3 \cdot Et_2O$ (0.3 mL) and triethyl orthoformate (0.3 mL) were added, and the mixture was stirred for 1 h under nitrogen. Chloranil (0.71 g, 2.9 mmol) was added and the solution was refluxed for 45 min. The solvent was partly evaporated and the reaction mixture was purified by column chromatography on basic alumina. The first fraction (bright green product **2**) was eluted with CH_2Cl_2 and was subjected to a second chromatography (basic alumina, CH_2Cl_2). The product was recrystalized from CH_2Cl_2 with CH_3OH or hexane to yield **2** as a dark green solid in a yield of 11%. UV/Vis (CH_2Cl_2): $\lambda_{\max}$ ($\log\varepsilon$) = 348 (4.4), 464 (4.5), 668 nm (4.2); for **2**-H_2^{2+} (with TFA): $\lambda_{\max}$ ($\log\varepsilon$) = 337 (4.4), 457 (sh), 490 (4.9), 767 (4.5), 825 nm (sh); ^{1}H NMR (500 MHz, $CDCl_3$, 210 K): $\delta = 8.53$ (s 2H, tell., 12,13), 8.10 (d, ^3J(H,H) = 7.3 Hz, 4H, *o*-Ph), 8.00 (d, ^3J(H,H) = 4.5 Hz, 2H, pyrr), 7.74 (d, ^3J(H,H) = 7.3 Hz, 2H, *o*-Ph), 7.65 (d, ^3J(H,H) = 7.1 Hz, 2H, *o*-Ph), 7.56 (m, 14H, pyrr) 7.59 (d, ^3J(H,H) = 4.5 Hz, *m,p*-Ph), 6.08 (s, 2H, 2H,3H-tell.); for **2**-H_2^{2+} (20 equiv TFA): $\delta = 9.03$ (d, ^3J(H,H) = 7.2 Hz, 4H, *o*-Ph), 8.71 (d, ^4J(H,H) = 1.5 Hz, 4H, pyrr), 8.09 (t, 2H, *m*-Ph), 8.03 (s, 4H, tell.), 7.95 (m, 12H, *o,m,p*-Ph), −0.70 (brs, 2H, NH); HR-MS: *m/z*: 844.0391; *m/z* calcd for $C_{44}H_{28}N_2{}^{130}Te_2$: 844.0381; elemental analysis: calcd for $C_{44}H_{28}N_2Te_2 \cdot 2CH_3OH$: C 61.12, H 4.01, N 3.10; found: C 61.13, H 3.89, N 3.26.

Received: July 12, 2001 [Z17488]



Scheme 2. Regular (**R**) and flipped (**F**) forms of Te_2TPP in conformational equilibrium.

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[15] Data collection: the measurement was performed on a Kuma KM4CCD diffractometer using $\mathrm{Mo}_{K\alpha}$ radiation ($\lambda = 0.71073$ Å), $T = 90$ K, in ω scan mode, $2\theta_{max} = 50$. **2**: crystals were prepared by the slow diffusion of CH_3OH into a solution of **2** in CH_2Cl_2 to yield a dark green crystal of $C_{44}H_{28}N_2Te_2$, crystal dimensions $0.07 \times 0.07 \times 0.18$ mm^3, monoclinic, space group $P2_1/n$, $a = 11.579(2)$, $b = 12.609(2)$, $c = 23.035(3)$ Å, $\beta = 99.11(1)°$, $V = 3320.8(8)$ Å^3, $\rho_{calcd} = 1.680$, $Z = 4$; total no. of reflections collected: 18630; no. of independent reflections: 5824 of which 3988 were included in the refinement of 433 parameters; an absorption correction based on a least-squares fit against $|F_c| - |F_o|$ differences was applied:[18] $\mu = 1.793$ mm^{-1}, $A_{min.} = 0.8456$, $A_{max.} = 1.21949$. The structure was solved by using direct methods with SHELXS-97 and refined against $|F^2|$ using SHELXL-97 (G. M. Sheldrick, University of Göttingen, Germany, **1997**), final R_1/wR_2 indices (for $I > 2\sigma(I)$): 0.0810/0.0662; max./min. residual electron density: $+0.716/-0.597$ e A^{-3}; H atoms were fixed. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-166826. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

[16] The X-ray crystal structure of $Te_2TPP \cdot CHCl_3$, although of poor quality, confirmed the shape of **2**.

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Remarkable Rate Enhancement of Ligand Substitution Promoted by Geometrical Arrangement of Tridentate "Spectator" Ligands[**]

My Hang V. Huynh,* Jennifer Smyth, Modi Wetzler, Brendan Mort, Paul K. Gong, Laura M. Witham, Donald L. Jameson, David K. Geiger, Joanne M. Lasker, Mina Charepoo, Michelle Gornikiewicz, Jose M. Cintron, Gretchen Imahori, Roberto R. Sanchez, Amy C. Marschilok, Lynn M. Krajkowski, David G. Churchill, Melvyn Rowen Churchill, and Kenneth J. Takeuchi*

Dedicated to Dr. Thomas J. Meyer
on the occasion of his 60th birthday

The reactivity exhibited by transition metal complexes depends on the oxidation state of the metal center[1] and on stereoelectronic (steric and electronic) effects of the ligands.[2] Since the initial development of poly(pyrazol-1-yl)alkanes[3] and poly(pyrazol-1-yl)borates[4] by Trofimenko, these so-called "scorpionate" ligands have been used in coordination, organometallic, and bioinorganic chemistry.[5–6] Utilizing these scorpionate ligands and different geometrical arrangements of tridentate "spectator" ligands, we observe a dramatic 1.9×10^7 rate increase for ligand substitution at a supposedly inert ruthenium(II) center. To explain these unexpected results, ligand substitution kinetics for complexes having different ligand geometries are compared. Scheme 1 depicts two systems, one in which the active site lies in the plane of the

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[**] K.J.T. acknowledges Arco Chemical and the National Science Foundation for support of this research. M.H.V.H. gratefully acknowledges postdoctoral fellowship support from the Director's Office of Los Alamos National Laboratory. Los Alamos National Laboratory is operated by the University of California for the U.S. Department of Energy under Contract W-7405-ENG-36. M.H.V.H. also thanks Dr. Beverly K. Hartline (Deputy Laboratory Director, Argonne National Laboratory), Dr. Donald G. Lee (Chemistry Professor, University of Regina, Saskatchewan, Canada, S4S0A2), Dr. R. Thomas Baker (Los Alamos National Laboratory), and Dr. David E. Morris (Los Alamos National Laboratory) for their suggestions and discussions.

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