

- anisotropic displacement parameters: 897 parameters,  $wR2 = 0.1835$  for 18516 unique data ( $2.69 \leq \theta \leq 26.39^\circ$ ),  $R1 = 0.0666$  for 14232 data with  $F_o > 4\sigma(F)$ , max./min. residual electron density 0.789/−0.549 e Å<sup>−3</sup>. 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-148156. 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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## Off-the-Shelf Colorimetric Anion Sensors\*\*

Hidekazu Miyaji and Jonathan L. Sessler\*

The recognition<sup>[1]</sup> and sensing<sup>[2]</sup> of anionic analytes has emerged recently as a key research theme within the generalized area of supramolecular chemistry.<sup>[3]</sup> Of particular interest in this regard are “colorimetric anion sensors”, species that would allow the so-called “naked-eye” detection of anions without resort to any spectroscopic instrumentation. However, few such systems exist at present.<sup>[4–7]</sup> For instance, alizarin complexone<sup>[4a]</sup> has been used to effect the colorimetric sensing of fluoride anions in water, wet acetone, and wet acetonitrile as has 2,3-dipyrrol-2-yl-quinoxaline in dichloro-

methane.<sup>[6b]</sup> Thiourea has also been used to sense halide, phosphate, and carboxylate ions.<sup>[4b]</sup> Appropriately functionalized calix[4]pyrroles also show potential as naked-eye anion sensors.<sup>[7]</sup> Separately, displacement-based strategies have been used by Lavigne and Anslyn to detect citrate anions colorimetrically<sup>[5]</sup> and, more recently by Gale et al. to sense halide anions.<sup>[6a]</sup> Still, there remains a cogent need for species whose changes in color could be used to signal either qualitatively or quantitatively the presence of particular anionic analytes. Such agents would be especially valuable were they to be obtained readily without resort to dedicated synthesis. Herein we report that a wide range of commercially available entities, such as 1,2-diaminoanthraquinone, 1,8-diaminoanthraquinone, 4-nitroaniline, 4-nitro-1,2-phenylenediamine, L-leucine-4-nitroanilide, 1-(4-nitrophenyl)-2-thiourea, 4-nitrophenol, alizarin, 2,2'-bi(3-hydroxy-1,4-naphthoquinone), acid blue 45, naphthol AS, 9(10*H*)-acridone, and Direct Yellow 50 may be used as “off-the-shelf” color-based anion sensors in organic solvent. We also show that some of these agents may be used in combination with a crown ether phase-transfer catalyst to extract and detect anions colorimetrically in a two-phase organic–aqueous set-up, even in those instances where the anions being detected were originally present in the aqueous phase.

Initially, we focused on 1,8-diaminoanthraquinone as a potential off-the-shelf anion sensor. We were attracted to this system because 1) it contains two aniline-like NH<sub>2</sub> donor groups that appeared properly positioned to bind a chloride ion by cooperative hydrogen-bonding interactions, as judged from structural analyses of previous chloride-binding<sup>[8]</sup> and noncolorimetric sensing agents<sup>[2c]</sup> containing a similar structural motif, 2) such binding was expected to result in a bathochromic shift in the absorption bands in the visible region,<sup>[7b]</sup> and 3) various control compounds, with other modifications of the anthraquinone skeleton, would be readily available. In accord with the latter predicative thought, we studied the changes (if any) in the absorption spectra of anthraquinone, 1,2-, 1,4-, 1,5-, 1,8-,<sup>[8]</sup> and 2,6-diaminoanthraquinone, as well as 1- and 2-aminoanthraquinone, induced upon the addition of various anions, namely F<sup>−</sup>, Cl<sup>−</sup>, Br<sup>−</sup>, I<sup>−</sup>, H<sub>2</sub>PO<sub>4</sub><sup>−</sup>, and HSO<sub>4</sub><sup>−</sup> in dichloromethane. Although, no change in the absorption spectrum of anthraquinone itself was found upon the addition of any of the analytes tested, the spectra of the other anthraquinone derivatives (those containing at least one hydrogen bond donor group) were all found to be modified in the presence of tetrabutylammonium fluoride. On the other hand, only the spectra of 1,2- and 1,8-anthraquinone were seen to undergo a discernable change when exposed to tetrabutylammonium chloride in this same solvent. Presumably, the fluoride ion, in contrast to the chloride ion, is a strong enough hydrogen-bond acceptor that it can interact with anthraquinone derivatives that contain only a single hydrogen-bond donor group or single accessible donor group.

Significant bathochromic shifts in the absorption spectra were also observed for 1,2- and 1,8-diaminoanthraquinone in dichloromethane in the presence of chloride, bromide, and phosphate ions. These spectral changes were particularly dramatic in the case of 1,2-diaminoanthraquinone, being

[\*] Prof. J. L. Sessler, Dr. H. Miyaji  
Department of Chemistry and Biochemistry  
and Institute for Cellular and Molecular Biology  
The University of Texas at Austin  
Austin, TX 78712–1167 (USA)  
Fax: (+1) 512-471-7550  
E-mail: sessler@mail.utexas.edu

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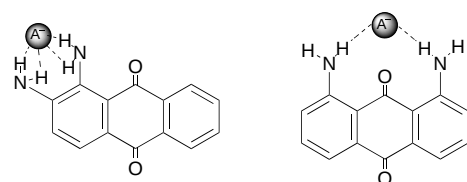
sufficiently large that this species was found to function as an effective colorimetric anion sensor. Figure 1 shows the color changes induced upon the addition of various anions (100 equiv each) to solutions of 1,2-diaminoanthraquinone in dichloromethane. Specifically, it was found that solutions,



Figure 1. Photograph showing the color of dilute solutions of 1,2-diaminoanthraquinone in dichloromethane ( $[\text{quinone}] = 1.0 \times 10^{-4} \text{ M}$ ). A) Before addition of the anion. After addition of 100 equivalents of B) fluoride, C) chloride, D) bromide, E) iodide, F) phosphate, and G) sulfate ions. All anions were added in the form of their tetrabutylammonium salts.

initially yellow in color ( $\lambda_{\text{max}} = 478 \text{ nm}$ ), became dark purple ( $\lambda_{\text{max}} = 555 \text{ nm}$ ), red ( $\lambda_{\text{max}} = 519 \text{ nm}$ ), reddish orange ( $\lambda_{\text{max}} = 513 \text{ nm}$ ), orange ( $\lambda_{\text{max}} = 499 \text{ nm}$ ), purple ( $\lambda_{\text{max}} = 548 \text{ nm}$ ), and orange ( $\lambda_{\text{max}} = 493 \text{ nm}$ ) when exposed to fluoride, chloride, bromide, iodide, phosphate, and sulfate ions, respectively.<sup>[9]</sup>

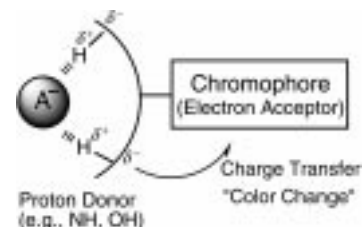
Solutions of 1,8-diaminoanthraquinone in dichloromethane were also found to change color when exposed to these anions, albeit to a lesser extent.<sup>[10]</sup> On the other hand, no significant changes in color were observed when solutions of 1,4-, 1,5-, 2,6-diaminoanthraquinone or 1- and 2-aminoanthraquinone in dichloromethane were exposed to 100 equivalents of chloride, bromide, iodide, phosphate, or sulfate ions. Such findings are consistent with the proposal that less basic anions, such as  $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{H}_2\text{PO}_4^-$ , and  $\text{HSO}_4^-$ , are not bound to an appreciable extent by a single  $\text{NH}_2$  site but do interact cooperatively with two appropriately spaced  $\text{NH}_2$  donor moieties (that is, in the 1,2 and 1,8 positions of anthraquinone). Although not studied in detail, the color change itself is ascribed to the formation of a charge-transfer complex as the result of these proposed solution-phase hydrogen-bonding interactions (Scheme 1).



Scheme 1. Proposed modes of anion binding for 1,2-diaminoanthraquinone and 1,8-diaminoanthraquinone.

Although hydrogen-bonding interactions are considered key to the success of the off-the-shelf anion sensing demonstrated by 1,2- and 1,8-diaminoanthraquinone, and while the initial choice of solvent, dichloromethane, was chosen to maximize these interactions, it was found that a discernable color-based response was observed when more polar solvents, namely  $\text{CH}_3\text{CN}$  and  $\text{DMSO}$ , were used.

The results obtained with 1,2- and 1,8-diaminoanthraquinone, led to the consideration that many species should be able to function as off-the-shelf anion sensors, provided that they possess hydrogen bond donor functionality, preferably two or more  $\text{-NH}_2$ ,  $\text{-C(O)NHR}$ , or  $\text{OH}$ -derived binding sites, and a chromophore subunit whose electronic properties are modified as the result of interaction with a bound anionic substrate (through, for example, electrostatic or charge transfer effects; see Scheme 2). In accord with the above thinking,



Scheme 2. Schematic representation illustrating the origins of the generalized "off-the-shelf" approach to anion sensing being proposed here.

many commercially available compounds were screened as possible off-the-shelf anion sensors. A listing of compounds showing readily discernable changes is given as Scheme 3 while Figure 2 illustrates representative color changes observed using some of these species.

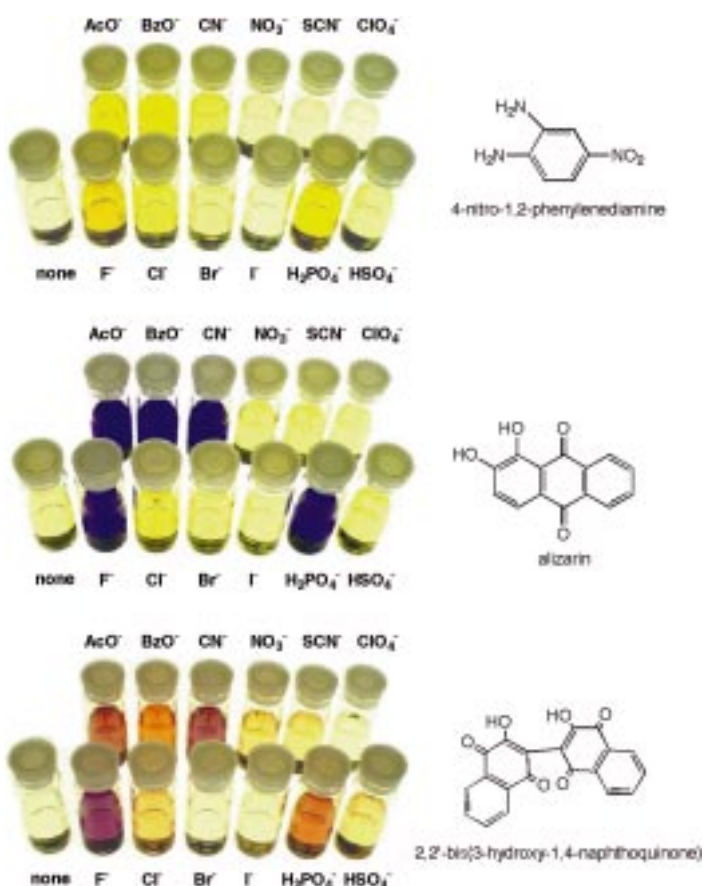


Figure 2. Color changes observed before and after dilute ( $1 \times 10^{-4} \text{ M}$ ) solutions of 4-nitro-1,2-phenylenediamine, alizarin, and 2,2'-bis(3-hydroxy-1,4-naphthoquinone) in dichloromethane were treated with 100 equivalents of the indicated anionic analytes. All anions were added in the form of their tetrabutylammonium salts.

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| <b>4-nitroaniline</b><br>(Colorless → Yellow / DCM)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | <b>4-nitro-1,2-phenylenediamine</b><br>(Pale Yellow → Orange ~ Yellow / DCM) | <b>L-leucine-4-nitroanilide</b><br>(Colorless → Yellow / DCM)                     |                                     |                                             |                                             |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| <b>4-nitrophenol</b><br>(Colorless → Yellow / DCM)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>alizarin</b><br>(Yellow → Blue ~ Purple / DCM)                            | <b>2,2'-bis(3-hydroxy-1,4-naphthoquinone)</b><br>(Yellow → Purple ~ Orange / DCM) |                                     |                                             |                                             |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| <b>1-(4-nitrophenyl)-2-thiourea</b><br>(Pale Yellow → Orange ~ Yellow / DMSO)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | <b>acid blue 45</b><br>(Blue → Colorless / DMSO)                             |                                                                                   |                                     |                                             |                                             |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| <b>naphthol AS</b><br>(Colorless → Fluorescent Yellow / DCM)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | <b>9(10H)-acridone</b><br>(Colorless → Fluorescent Yellow / DMSO)            | <b>direct yellow 50</b><br>(Yellow → Red ~ Orange / DMSO)                         |                                     |                                             |                                             |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| F <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Cl <sup>-</sup>                                                              | Br <sup>-</sup>                                                                   | I <sup>-</sup>                      | H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> | HSO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
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| AcO <sup>-</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | BzO <sup>-</sup>                                                             | CN <sup>-</sup>                                                                   | NO <sub>3</sub> <sup>-</sup>        | SCN <sup>-</sup>                            | ClO <sub>4</sub> <sup>-</sup>               |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |
| <input type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | <input type="checkbox"/>                                                     | <input checked="" type="checkbox"/>                                               | <input type="checkbox"/>            | <input type="checkbox"/>                    | <input type="checkbox"/>                    |                               |                                     |                                     |                          |                          |                                     |                          |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                          |                          |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                |                 |                 |                |                                             |                               |                                     |                                     |                                     |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                |                 |                 |                |                                             |                               |                                     |                                     |                          |                          |                                     |                                     |                  |                  |                 |                              |                  |                               |                                     |                                     |                                     |                                     |                                     |                          |

Scheme 3. Examples of commercially available colorimetric anion sensors. A checked box indicates a “naked-eye” detectable change in color is observed in the indicated solvents upon addition of 100 equivalents of the anionic analyte in question. DCM = dichloromethane; DMSO = dimethyl sulfoxide.

Based on the results summarized in Scheme 3, we believe that the use of appropriate off-the-shelf molecular entities could provide a generalized new approach to anion sensing. While lacking the specificity inherent in more sophisticated approaches to anion detection (for example, those based on specific receptor design), the fact that some inherent discrimination between anionic analytes is observed, coupled with its low cost, leads us to suggest that this new approach could find application in a variety of end-use areas, including multi-

analyte array construction,<sup>[11]</sup> where ease of use and cost of goods are important considerations.

In support of the above contention, 2,2'-bis(3-hydroxy-1,4-naphthoquinone)<sup>[12]</sup> was used as a potential chloride sensor under extraction-type conditions. As illustrated in Figure 3, it was found that in a two-phase, aqueous–dichloromethane system containing [15]crown-5 as a phase-transfer catalyst present in the organic phase acts as a colorimetric indicator of chloride anions originally present in the aqueous layer.<sup>[13]</sup> This

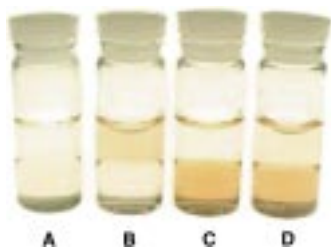


Figure 3. Color changes observed upon exposing aqueous solutions of chloride ions to dichloromethane phases containing 2,2'-bis(3-hydroxy-1,4-naphthoquinone) ( $1 \times 10^{-3}$  M). A) Control experiment. The aqueous phase is distilled water (pH 7.41). B) The aqueous phase contains NaCl at a concentration of  $1 \times 10^{-1}$  M. C) The aqueous phase contains NaCl ( $1 \times 10^{-1}$  M) and benzo[15]crown-5. D) The aqueous phase is Gulf of Mexico sea water (pH 7.34) and benzo[15]crown-5.

ability to sense qualitatively in a naked-eye-detectable fashion chloride anions is not limited to laboratory samples but was also found to work for sea water obtained from the Gulf of Mexico.<sup>[14]</sup> This observation leads us to suggest that this particular species could prove useful as a chloride anion sensor.<sup>[15]</sup>

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## Resolution and Kinetic Stability of a Chiral Supramolecular Assembly Made of Labile Components\*\*

Andreas J. Terpin, Marco Ziegler, Darren W. Johnson, and Kenneth N. Raymond\*

The importance of chirality for recognition processes in nature is impressively exemplified by the different tastes of (*R*)- and (*S*)-asparagine,<sup>[1]</sup> and the vastly different pharmacological effects of the two enantiomers of thalidomide.<sup>[2]</sup> Chirality is not the exclusive domain of organic chemistry, many metals can also serve as centers of chirality.<sup>[3]</sup> The most frequent case is an octahedral arrangement of three bidentate ligands around a metal center to form  $\Delta$  or  $\Lambda$  absolute configurations. Chirality at metal centers has been found to play an important role in nature, for example, for the siderophore-mediated iron uptake in many organisms.<sup>[4]</sup>

$\Delta/\Lambda$  Isomerization of tris-bidentate complexes of the labile high-spin ferric ion or its closely related gallium(III) analogues is rapid in aqueous solution. Tris(catecholate)gallium(III) complexes, model compounds of iron siderophores, usually racemize fast. Racemization rates of  $10(1) s^{-1}$  for a mononuclear complex<sup>[5]</sup> and  $0.10(1) s^{-1}$  for a dinuclear helix<sup>[6]</sup> were determined by NMR studies. The isomerization was found to

[\*] Prof. K. N. Raymond, A. J. Terpin, M. Ziegler, D. W. Johnson  
Department of Chemistry  
University of California  
Berkeley, CA 94720-1460 (USA)  
Fax: (+1) 510-486-5283  
WWW: <http://www.cchem.berkeley.edu/~knrgpr>  
E-mail: [raymond@socrates.berkeley.edu](mailto:raymond@socrates.berkeley.edu)

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