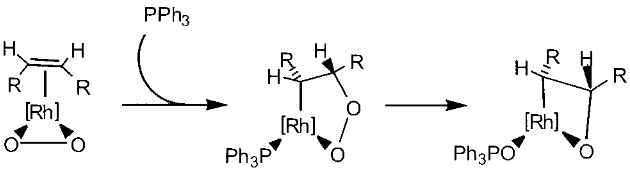
3-Rhoda-1,2-dioxolanes through Dioxygenation of a Rhodium – Ethene Complex by Air[**]

Monique Krom, Ruud G. E. Coumans, Jan M. M. Smits, and Anton W. Gal*

3-Metalla-1,2-dioxolanes (κ^2C^1,O^2-2-peroxyethyl metal complexes) have been proposed as intermediates in the catalytic oxidation of olefins by Group VIII metals.[1] These reactions include epoxidation[1d] and oxidation to ketones.[1a,c]

Read has invoked contraction of 3-rhoda-1,2-dioxolanes to 2-rhodaoxetanes by O atom transfer to triphenylphosphane in catalytic co-oxygenation of olefins and triphenylphosphane (Scheme 1).[1b]

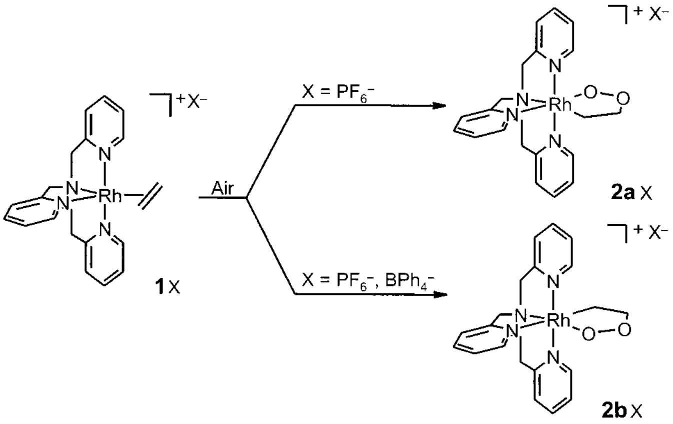


Scheme 1. 3-Rhoda-1,2-dioxolane and 2-rhodaoxetane intermediates in the co-oxygenation of triphenylphosphane and an olefin.[1b]

We have previously obtained the stable 2-rhodaoxetanes, [(tpa)Rh(κ^2C^1,O-CH_2CH_2O-)]$^+$ and [(Metpa)Rh(κ^2C^1,O-CH_2CH_2O-)]$^+$, by mono-oxygenation of the corresponding ethene complexes, [(tpa)Rh(CH_2CH_2)]$^+$ and [(Metpa)Rh(CH_2CH_2)]$^+$, with H_2O_2.[2] In these reactions an unsubstituted 2-rhodaoxetane fragment, [Rh(κ^2C^1,O-CH_2CH_2O-)]$^+$, is stabilized by the nitrogen donor ligands tpa and Metpa.[3] Analogous 3-metalla-1,2-dioxolanes, containing an unsubstituted 3-metalla-1,2-dioxolane fragment [M^n(κ^2C^1,O^2-CH_2CH_2OO-)]$^{(n-2)+}$, have not been reported to date. Tri- and tetrasubstituted 3-platina-1,2-dioxolanes, with β-carbon substituents that prevent β-hydrogen transfer, have been structurally characterized. They were obtained from the reaction of [$(PPh_3)_2Pt(\eta^2\text{-}O_2)$] with compounds containing tetrasubstituted electron-deficient double bonds.[4]

We now report the formation of a stable 3-metalla-1,2-dioxolane by dioxygenation of a metal olefin complex. The rhodium(I)-ethene fragment, [Rh(CH_2CH_2)]$^+$, stabilized by tpa, is found to be selectively dioxygenated by air to an unsubstituted 3-rhoda-1,2-dioxolane fragment. The structure of this dioxolane fragment has been determined by X-ray diffraction (see Figure 1).

The ethene complex [(tpa)Rh(CH_2CH_2)]PF_6 (**1**PF_6, Scheme 2) was synthesized according to a literature procedure.[2b] Oxidation of *a solution of* **1**PF_6 with air at room temperature results in loss of ethene from **1**$^+$ and formation of as yet unidentified products. In marked contrast, exposure of *solid* **1**PF_6 to air results in a mixture of the products **2a**PF_6 and **2b**PF_6 in an approximate ratio of 1:1.



Scheme 2. Oxidation of **1**$^+$ to 3-rhoda-1,2-dioxolanes. X = PF_6 (**1**, **2a**, **2b**), BPh_4 (**1**, **2b**).

Compounds **2a**PF_6, **2b**PF_6, and **2b**BPh_4 (see below) proved sensitive towards visible light and therefore had to be manipulated in the dark. The nature of their photochemical reaction products will be subject of a forthcoming publication.

Electrospray ionization mass spectrometry (ESI-MS) and ^{1}H NMR spectra of the mixture of **2a**PF_6 and **2b**PF_6 in CD_3CN indicate that **2a**$^+$ and **2b**$^+$ are the two isomeric 3-rhoda-1,2-dioxolanes shown in Scheme 2. The base peak at $m/z = 453$ in the ESI-MS spectrum is consistent with the presence of [(tpa)Rh(CH_2CH_2) + O_2]$^+$. The couplings $^3J(H_\alpha,H_\beta)$ of the RhCH_2CH_2- fragments in **2a**$^+$ and **2b**$^+$ (6.1 Hz, Table 1) suggest them to be part of a five-membered ring: $^3J(H_\alpha,H_\beta)$ decreases from 7.5 Hz for the four-membered 2-rhodaoxetane ring in **3a**$^+$,[2a,b] to 6.1 Hz for the five membered ring in **2a**$^+$ and **2b**$^+$, to only 5.6 Hz for the six-membered ring in rhodium 2-(acetimidoyloxy)ethyl complex **4a**$^+$ (Scheme 3).[2a] The nuclear Overhauser enhancement (NOE) interactions in **2a**$^+$ and **2b**$^+$ are consistent with their proposed structures: isomer **2b**$^+$ shows NOE interactions between Rh$\mathit{CH_2}$$CH_2$- and one proton each of the two equivalent Rh-N_{amine}-$\mathit{CH_2}$- units, and between Rh$CH_2$$\mathit{CH_2}$- and the two equivalent pyridyl (py) H6 centers; isomer **2a**$^+$ shows NOE interactions between Rh$\mathit{CH_2}$$CH_2$- and all three pyridyl H6 centers, and between Rh$CH_2$$\mathit{CH_2}$- and the two equivalent pyridyl H6 centers.

In an attempt to selectively obtain one of the two isomeric 3-rhodadioxolanes **2a**$^+$ and **2b**$^+$, we changed the counterion of **1**$^+$ from PF_6^- to BPh_4^-.[5] Upon reaction of *solid* **1**BPh_4 with air only **2b**BPh_4 was formed, as confirmed by the ^{1}H NMR and ESI-MS spectra. Possibly, different packings in (microcrystalline) **1**BPh_4 and **1**PF_6 have resulted in a different accessibility of O_2 to the ion **1**$^+$. Remarkably, from the oxidation of **1**$^+$ by hydrogen peroxide only the 2-rhodaoxetane **3a**$^+$, the analogue of isomer **2a**$^+$, has been obtained

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(Scheme 3). Thus far, a 2-rhodaoxetane analogue of isomer **2b**$^+$ has not been obtained.

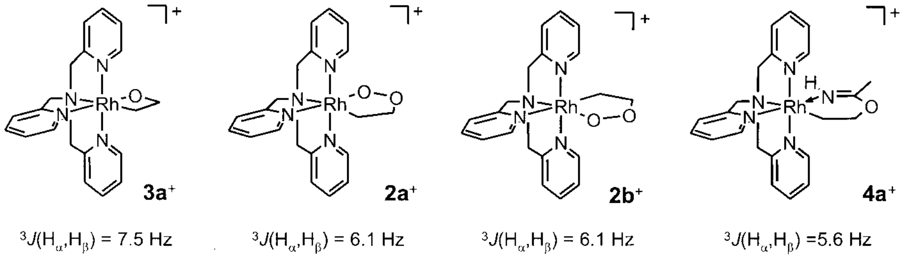
The structure of complex **2b**BPh_4, as determined by X-ray diffraction, is shown in Figure 1.[6] The 5-membered ring has the envelope conformation and occurs in two almost mirrored, but crystallographically independent, conformations (**2b**A^+ and **2b**B^+). The bond Rh$-$N$_{amine}$ (N1-Rh1) in 3-rhoda-1,2-dioxolane **2b**$^+$ is shorter than that found in an analogue of 2-rhodaoxetane **3a**$^+$ (Scheme 3; 2.056(2) Å vs. 2.132(8) Å av.);[2a,b] as expected, the *trans* influence of a 3-rhoda-1,2-dioxolane O_α is smaller than that of a 2-rhoda-oxetane C_α. The puckering of the 3-rhoda-1,2-dioxolane ring in the cations **2b**A^+ and **2b**B^+ is virtually the same as found in the α-dicyano-β-dimethyl substituted 3-platina-1,2-dioxolane ring.[4a, 7] The average O1$-$O2 bond in **2b**A^+ and **2b**B^+ (1.481(17) Å) is normal when compared to the O$-$O bond in the 3-platina-1,2-dioxolane (1.482(15) Å)[4a] and other M-O-O-C compounds (1.40 – 1.52 Å).[8] The average distance O2$-$C2 for **2b**A^+ and **2b**B^+ (1.438(11) Å) is similar to the O$-$C distances in the 3-platina-1,2-dioxolane (1.407(19) Å) and in other M-O-O-C compounds (1.40 – 1.47 Å).[9]

We have thus for the first time selectively oxidized a metal – ethene complex with air to a stable 3-metalla-1,2-dioxolane. Selective dioxygenation of **1**$^+$ was successful only for **1**$^+$ in a crystalline matrix. The observed formation of stable isomeric 3-rhoda-1,2-dioxolanes from a rhodium – ethene complex lends support to the involvement of 3-rhoda-1,2-dioxolanes as intermediates in rhodium-catalyzed oxygenation of olefins to ketones, as proposed by Mimoun et al.[1a] and Read.[1b] Both the mechanism of the observed selective oxidation in the solid state and the reactivity of the resulting 3-rhoda-1,2-dioxolanes are currently under investigation.

Table 1. Chemical shifts (ppm) and coupling constants (Hz) of the $RhCH_2CH_2O$- fragments in CD_3CN.

	$\delta(RhCH_2)$	$\delta(CH_2O)$	$^3J(H,H)$	$^2J(H,Rh)$	$\delta(RhCH_2)$	$\delta(CH_2O)$	$^1J(C,Rh)$
3a$^+$	2.24(dt)	4.88(t)	7.5	2.5	1.3(d) [a]	78.7(d)	18.4[a]
2a$^+$	3.55(dt)	3.41(t)	6.1	2.4	44.8(d)	72.8(s)	28.8
2b$^+$	2.57(dt)	3.25(t)	6.1	2.4	35.0(d)	70.2(s)	25.0

[a] $[D_6]$Acetone.



Scheme 3. Decrease of $^3J(H_\alpha,H_\beta)$ with increasing ring size.

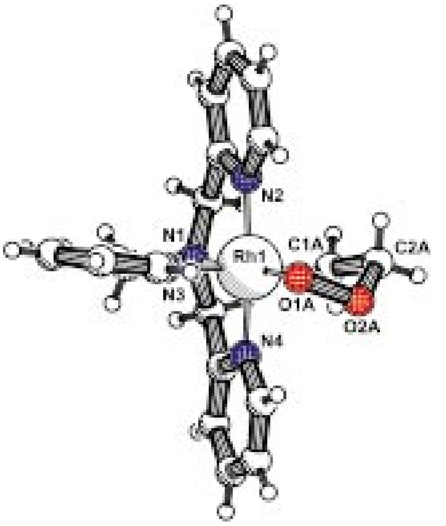


Figure 1. X-ray structure of **2b**BPh_4; only one of the two independent cations is shown (**2b**A^+, but data for **2b**B^+ are also listed) . Selected bond lengths [Å]: Rh1-N1 2.056(2), Rh1-N2 2.015(2), Rh1-N3 2.144(3), Rh1-N4 2.025(2), Rh1-C1A 2.05(3), Rh1-C1B 2.06(4), Rh1-O1A 1.99(3), Rh1-O1B 2.00(3), O1A-O2A 1.481(17), O1B-O2B 1.481(16), O2A-C2A 1.431(11), O2B-C2B 1.445(11) C1A-C2A 1.548(13), C1B-C2B 1.531(15).

Experimental Section

NMR experiments were carried out on Bruker DPX200, Bruker AC300, and Bruker AM500 spectrometers. Solvent shift references for NMR are: CD_3CN $\delta(^1H) = 1.94$, $\delta(^{13}C) = 118.1$. ESI-MS spectra were recorded on a Finnigan MAT 900S mass spectrometer.

Compounds $[(tpa)Rh(CH_2CH_2)]BPh_4/PF_6$,[2b] $[\{(CH_2CH_2)_2RhCl\}_2]$,[10] and tpa[11] were prepared according to literature procedures.

2aPF_6/**2b**PF_6: **1**PF_6 was powdered with a mortar and exposed to air at room temperature and in the dark for 2 days at atmospheric pressure. A mixture of **2a**PF_6 and **2b**PF_6 in an approximate ratio of 1:1 was obtained in >90 % yield, as determined by ^1H NMR. The data for **2a**PF_6 are: ^1H NMR (500 MHz, CD_3CN, 298 K): $\delta = 8.86$ (d, 1 H, $^3J(H,H) = 5.4$ Hz, py$_a$-H6), 8.56 (d, 2 H, $^3J(H,H) = 5.4$ Hz, py$_b$-H6), 7.83 (dt, 2 H, $^3J(H,H) = 7.8$ Hz, $^4J(H,H) = 1.4$ Hz, py$_b$-H4), 7.63 (t, 1 H, $^3J(H,H) = 7.3$ Hz, py$_a$-H4), 7.48 (d, 2 H, $^3J(H,H) = 7.8$ Hz, py$_b$-H3), 7.35 (t, 2 H, $^3J(H,H) = 6.6$ Hz, py$_b$-H5), 7.25 (m, 1 H, py$_a$-H5), 7.14 (d, 1 H, $^3J(H,H) = 7.8$ Hz, py$_a$-H3), 5.12 (d[AB], 2 H, $^2J(H,H) = 15.6$ Hz, NCH_2-py$_b$), 5.00 (d[AB], 2 H, $^2J(H,H) = 15.6$ Hz, NCH_2-py$_b$), 4.80 (s, 2 H, NCH_2-py$_a$), 3.55 (dt, 2 H, $^3J(H,H) = 6.1$ Hz, $^2J(H,Rh) = 2.4$ Hz, RhCH_2CH$_2$O), 3.41 (t, 2 H, $^3J(H,H) = 6.1$ Hz, RhCH$_2$CH_2O); $^{13}C\{^1H\}$ NMR (50 MHz, CD_3CN, 300 K): $\delta = 164.6$ (py$_b$-C2), 162.7 (py$_a$-C6), 152.5 (py$_a$-C6), 150.5 (py$_b$-C6), 139.7 (py$_b$-C4), 138.7 (py$_a$-C4), 125.9 (py$_b$-C3), 125.5 (py$_a$-C3), 124.6 (py$_b$-C5), 122.6 (py$_a$-C5), 72.8 (RhCH$_2$*C*HO), 67.9 (N*C*H$_2$-py$_b$), 65.4 (N*C*H$_2$-py$_a$), 44.8 (d, $^1J(C,Rh) = 28.8$ Hz, Rh*C*H$_2$CH$_2$O); ESI-MS (CD_3CN): 453 $[M - PF_6]^+$, 425 $[M - C_2H_4 - PF_6]^+$, 409 $[M - C_2H_4O - PF_6]^+$, 393 $[M - C_2H_4O_2 - PF_6]^+$, 391 $[M - C_2H_4O_2 - H_2 - PF_6]^+$.

2bBPh_4: **1**BPh_4 was powdered with a mortar and exposed to air at room temperature and in the dark for 2 days at atmospheric pressure. **2b**BPh_4 was obtained in >90 % yield, as determined by ^1H NMR spectroscopy. ^1H NMR (300 MHz, CD_3CN, 297 K): $\delta = 8.75$ (d, 1 H, $^3J(H,H) = 5.4$ Hz, py$_a$-H6), 8.66 (d, 2 H, $^3J(H,H) = 5.5$ Hz, py$_b$-H6), 7.81 (dt, 2 H, $^3J(H,H) = 7.8$ Hz, $^4J(H,H) = 1.7$ Hz, py$_b$-H4), 7.69 (dt, 1 H, $^3J(H,H) = 7.8$ Hz, $^4J(H,H) = 1.7$ Hz, py$_a$-H4), 7.40 (d, 2 H, $^3J(H,H) = 8.0$ Hz, py$_b$-H3), 7.35 (m, 3 H, py$_a$-H5 and py$_b$-H5), 7.28 (m, 8 H, BAr-H2), 7.16 (dt, 1 H, $^3J(H,H) = 7.8$ Hz, $^4J(H,H) = 0.9$ Hz, py$_a$-H3), 6.99 (t, 8 H, $^3J(H,H) = 7.4$ Hz, BAr-H3), 6.84 (t, 4 H, $^3J(H,H) = 7.3$ Hz, BAr-H4), 4.88 (d[AB], 2 H, $^2J(H,H) = 15.6$ Hz, NCH_2-py$_b$), 4.71 (d[AB], 2 H, $^2J(H,H) = 15.1$ Hz, NCH_2-py$_b$), 4.57 (s, 2 H, NCH_2-py$_a$), 3.25 (t, 2 H, $^3J(H,H) = 6.1$ Hz, RhCH$_2$CH_2O), 2.57 (dt, 2 H, $^3J(H,H) = 6.1$ Hz, $^2J(H,Rh) = 2.4$ Hz, RhCH_2CH$_2$O-); $^{13}C\{^1H\}$ NMR (75 MHz, CD_3CN, 297 K): $\delta = 164.6$ (q, $^1J(C,B) = 49.5$ Hz, BAr-C1), 162.7 (py$_b$-C2), 158.6 (py$_a$-C2), 150.1 (py$_b$-C6), 149.5 (py$_a$-C6), 139.4 (py$_a$-C4), 139.3 (py$_b$-C4), 136.5 (BAr-C2), 126.4 (q, ^3J(C,B) = 2.8 Hz, BAr-C3), 125.5 (py$_b$-C3), 125.4 (py$_a$-C3), 124.2 (py$_b$-C5), 122.6 (BAr-C4), 121.9 (py$_a$-C5), 72.9 (NCH$_2$-py$_a$), 70.2 (NCH$_2$-py$_b$ and RhCH$_2$$CH_2$O), 35.0 (d, ^1J(C,Rh) = 25.0 Hz, RhCH$_2$CH$_2$O); ESI-MS (CD$_3$CN): 453 $[M-\mathrm{BPh_4}]^+$, 425 $[M-\mathrm{C_2H_4}-\mathrm{BPh_4}]^+$, 409 $[M-\mathrm{C_2H_4O}\text{-}\mathrm{BPh_4}]^+$, 393 $[M-\mathrm{C_2H_4O_2}-\mathrm{BPh_4}]^+$, 391 $[M-\mathrm{C_2H_4O_2}-\mathrm{H_2}-\mathrm{BPh_4}]^+$.

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[3] tpa = tri(2-pyridylmethyl)amine; Metpa = [(6-methyl-2-pyridyl)methyl]di(2-pyridylmethyl)amine.

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[5] NaBPh$_4$ was used instead of KPF$_6$ to precipitate the ethene complex.

[6] Transparent yellow crystals of **2b**BPh$_4$ were obtained by slow diffusion of diethylether into a 1,2-dichloroethane/acetonitrile solution. The X-ray diffraction data were collected at 150(2) K on an Enraf-Nonius CAD4 diffractometer with rotating anode and CCD area detector using graphite monochromatized Mo$_{K\alpha}$ radiation (λ = 0.71073 Å). The structure was solved by the PATTY[12] option of the DIRDIF[13] program system. Crystal data: $\mathrm{C_{48}H_{50.50}BClN_{4.50}O_{2.50}Rh}$, M_r = 879.60, monoclinic, space group $C2/c$, a = 35.8999(8), b = 10.7567(3), c = 24.2379(5) Å, β = 116.4699(12), V = 8378.6(3) Å^3, Z = 8, ρ_{calcd} = 1.395 Mg m^{-3}. All non-hydrogen atoms that could be located were refined with anisotropic temperature factors. The hydrogen atoms were placed at calculated positions and refined isotropically in riding mode. The 5-membered dioxolane ring has the envelope form and occurs in two conformations. As expected there is considerable positional overlap for some of the atoms (C1A and C1B, C2A and C2B, O1A and O1B). Therefore some geometrical constraints had to be applied: the two conformations were "SAMEd" and the anisotropic thermal displacement parameters were tied. Calculations (PLATON, Spek 1995)[14] showed two distinct voids, one of 228 Å^3, containing 68 electrons, around a two-fold axis (position 4e, 0, y, 1/4; y = −0.037), and one of 151 Å^3, containing 51 electrons, around an inversion center (position 4b: 1/2, 0, 0). Based on the synthetic route and evidence from NMR spectroscopy it is assumed that these electron densities possibly stand for one molecule diethylether ($\mathrm{C_4H_{10}O}$, 42 electrons) plus one molecule acetonitrile ($\mathrm{CH_3CN}$, 22 electrons) in the first void (64 electrons, 28.5 Å^3/atom), and one molecule dichloroethane ($\mathrm{C_2H_4Cl_2}$, 50 electrons) in the second void (30.2 Å^3/atom). It was not possible to assign any physically meaningful parameters to the electron densities found in the difference fourier map. Therefore the SQUEEZE procedure was applied to account for these electron densities. 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-157414. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

[7] O$_\beta$ and C$_\beta$ (O2 and C2) are positioned above and below the O1-M-C1 plane: the displacement of O2 from this plane is +0.51 Å in **2b**A$^+$ and −0.46 Å in **2b**B$^+$, compared to +0.49 Å in the 3-platina-1,2-dioxolane. The displacement of C2 is −0.21 Å in **2b**A$^+$ and +0.27 Å in **2b**B$^+$, compared to −0.29 in the 3-platinadioxolane.

[8] This range is based on O−O bonds in 48 crystal structures containing a M-O-O-C fragment (M = any metal); 4 outliers were not taken into account.

[9] This range is based on O−C bonds in 48 crystal structures containing a M-O-O-C fragment (M = any metal); 9 outliers were not taken into account.

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A Striking, Multifaceted, Decalithium Aggregate with Carbanion, Organoamide, and Alkoxide Functionalities[**]

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Mixed-anion alkali metal complexes are of significant interest because of their ability to act as superbases.[1] Normally superbase aggregates are composed of two anions and two differing metals, $[\mathrm{R'M\cdot M'OR}]_n$ (R′ = alkyl, aryl, amide; R = alkyl; M = Li; M′ = Na, K), and it is these complexes that display greatest reactivity and have the widest application in synthetic processes.[1d,e] Solid-state structural information on mixed-anion systems is of vital importance to provide a more complete understanding of the reasons behind their differing reactivity. However, such complexes have historically proved extremely difficult to crystallize,[1a, 2] hence solid-state structures are scarce despite the emergence of some data for each class of mixed-anion alkali metal complexes.[3]

Unimetallic mixed-anion aggregates (M = M′), though usually less effective, are also of significant synthetic importance since, with suitable anions and metal, they can also show increased reactivity over that of the two separate starting reagents, for example $[n\mathrm{BuLi\cdot LiO(CH_2)_2NMe_2}]$.[4]

Herein, we report the crystallization and structure determination of the remarkable multifeatured unimetallic complex $[\{\mathrm{N(SiMe_3)(2\text{-}(2'\text{-}C_6H_4O)C_6H_4)}\}_2\mathrm{Li_4\cdot LiOEt\cdot(Et_2O)}]_2\cdot$ hexane, **1** · hexane, which contains *three* different superbase anionic components, amide, carbanion, and alkoxide. The presence of *ethoxide* is particularly striking since Lochmann has indicated that the most stable mixed-anion aggregates are

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