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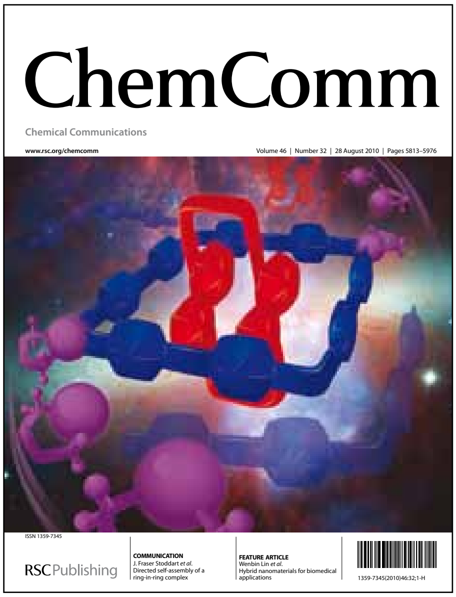

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COMMUNICATION

Catalytic "Triangles": Binding of Iron in Task-Specific Ionic Liquids†

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A new class of task-specific ionic liquids (ILs) which contain septuply positively charged $\{Fe^{III}_3O(RCOO)_6L_3\}^{7+}$ triangles has been synthesized and structurally characterized. Such metal-containing ILs can be repeatedly used as alternative catalysts in the synthesis of 2-pyrrolo-3'-yloxindole or the condensation of indoles with various aldehydes.

Salts consisting of ions and with melting points below 100˚C are called "Ionic Liquids" (ILs). They have receive attention as potential green solvents because they are non-flammable, non-volatile, possess good electrolytic properties with a large electrochemical window, have tunable polarity and are easy to recycle.[1] Also these salts may be designed for specific applications by incorporation of functionalities (*task-specific ionic liquids*)[2] in one or both ions as well as by the choice of cation–anion combination. Metal-ion-containing ILs represent a promising subclass of ILs since they may have interesting magnetic, luminescence or catalytic properties.[3] Apart from a few examples of ILs based on rare earth metal ions,[4] most investigations on metal-based ILs have been limited to transition metals (M^{Z+}) or Al^{3+} in $[MX_n]^{(n-z)-}$ anions containing compounds (where X = halide$^-$, SCN$^-$, Tf_2N^-, $(OC(CF_3)_3)^-$, $(F_6\text{-acac})^-$)[3d, 5] or as inorganic ionic liquids with polyoxometalate anions $[MTiW_{11}O_{39}]^{5-/6-}$.[6] A drawback of these compounds is their undesired properties such as poor solubility in water (or hydrolytic instability) and high viscosity. The examples of ionic liquids containing transition metal-based cations[7] are limited to derivatives of N-alkylimidazoles,[7b, 7c] R_1-NH-R_2 or metallocenium-based ligands, where R_1 or R_2 can be H, alkyl or –CH_2-CH_2-OH groups.[8] It has been widely demonstrated that the physical and chemical properties of ILs and their catalytic activity can be significantly influenced by the presence of small amounts of impurities. Therefore the challenge is to develop synthetic protocols which give high-purity ILs with the goal of utilizing the diversity of possible cation-anion combinations available. Herein we present the synthesis, structural characterization and catalytic properties of a new class of ionic liquids which contain septuply positively charged antiferromagnetically coupled $\{Fe^{III}_3O(RCOO)_6L_3\}^{7+}$ cationic triangles **2**-**6**. A survey of the Cambridge Crystallographic Database (CSD) up to Nov. 2011 reveals that more than 1100 crystal structures of metal carboxylates containing μ_3-oxo bridged metal triangle motifs have been deposited. However, so far the use of this motif in ILs is unprecedented. The new compounds were characterized by single-crystal X-ray diffraction, elemental analyses, IR, temperature-dependent thermal analyses (differential scanning calorimetry), susceptibility measurements and temperature-dependent Mössbauer spectra for compound **3**

Compounds **1**–**6** can be obtained in moderate yields via optimized synthetic procedures (Scheme S1, molecular structure of **1** in Fig. S1). The important solid-glass/liquid transition temperatures of compounds **1**–**6** (DSC) are presented in Table 1 (more detailed data in ESI†, Fig. S2). Quaternized salts **2**-**6** have ionic structures composed of $[Fe^{III}_3O(RCOO)_6(L)_3]^{7+}$ complex cations and a combination of $FeCl_4^-$, Cl^- and Tf_2N^- anions with water or methanol molecules as terminal ligands plus lattice water molecules. An example of the complex cation (for compound **2**) and seven $FeCl_4^-$ counter-anions is shown in Fig. 1.

Table 1. Melting Points/or Glass Transition Points (onset temperature, ˚C) and catalyst activity parameters of investigated compounds (formulas presented without solvent water molecules).

Compound	T1) (˚C)	Yield/time (%/hours)	
		CS1 2)	CS2 3)
$[mcmmim]FeCl_4$ (**1**)	42[b]	86/2	82/5
$[Fe_3O(cmmim)_6(H_2O)_3](FeCl_4)_7$ (**2**)	92[a]	94/2	92/5
$[Fe_3O(cmmim)_6(MeOH)_3](FeCl_4)_7$ (**3**)	-	-	-
$[Fe_3O(cmmim)_6(H_2O)_3](FeCl_4)_3Cl_4$ (**4**)	103[a]	98/2	85/5
$[Fe_3O(cmmim)_6(H_2O)_3](Tf_2N)_7$ (**5**)	92[b]	98/2	70/5
$[Fe_3O(cepy)_6(H_2O)_3](FeCl_4)_6Cl$ (**6**)	94[a]	96/2	89/5

1) T_g (glass transition point)[a]/$T_{m.p.}$ (melting point)[b] at heating (˚C); 2) synthesis of ethyl 2-methyl-4-(2-oxo-2,3-dihydro-1*H*-3-indolyl)-5-phenyl-1*H*-3-pyrrolecarboxylate **7a** using ~0.65 mol% of co-catalyst at RT; 3) synthesis of 3,3'-((4-methoxyphenyl)methylene)bis(1H-indole) **8a**; mcmmim ($C_1ImC_1CO_2CH_3$) ≡ methyl 2-(3-methyl-1H-imidazol-1-yl)acetate; cmmim ($C_1ImC_1CO_2$) ≡ 1-carboxymethyl-3-methylimidazolium; Tf_2N^- ≡ bis(trifluromethane)sulfonamide; cepy (PyC_2CO_2) ≡ N-(2-carboxyethyl)pyridinium.

At the centre of each triangle there is a μ_3-oxo oxygen atom connecting the three iron atoms (see ESI†, Table S1, Figures S3-S7). Each iron(III) has an octahedral O_6 coordination environment formed by the μ_3-oxo atom, four oxygens from the carboxylate ligands and the oxygen atom from the water ligand. Pairs of irons are further linked by double bidentate-bridging carboxylate groups with Fe-O distances between 1.99-2.06 Å.

Thus the structures are very similar to those of the "classic" trinuclear transition metal(III) carboxylate triangles.[9]

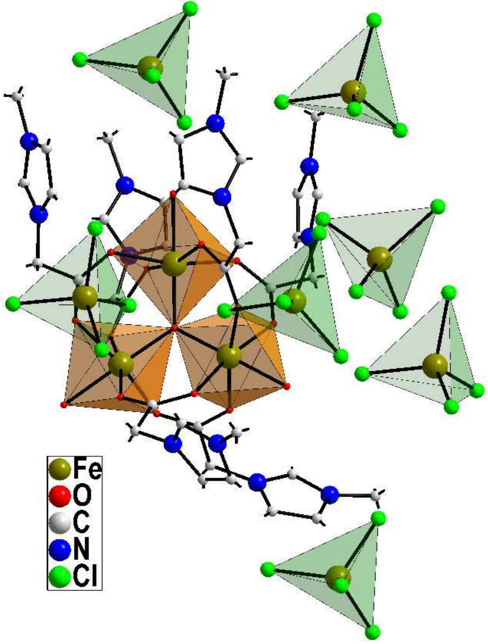


Figure 1. Molecular fragment for decanuclear "double" (cation-anion) iron based $[Fe_3O(C_1ImC_1CO_2)_6(H_2O)_3](FeCl_4)_7$ cluster (**2**). Hydrogen atoms and solvent water molecules were omitted for clarity. Polyhedra correspond to octahedral FeO_6 (brown) and tetrahedral $FeCl_4$ (green) environments.

The results of magnetic susceptibility measurements on the polycrystalline compound **3** are shown in Figure 2. For compound **3** the room temperature χT product is 34.75 $cm^3{\cdot}K{\cdot}mol^{-1}$, lower than the expected value (43.75 $cm^3{\cdot}K{\cdot}mol^{-1}$) for the presence of ten Fe^{III} ions (S = 5/2, g = 2). On decreasing the temperature, the χT product at 0.1T decreases smoothly until 30 K (30.43 $cm^3{\cdot}K{\cdot}mol^{-1}$) and then undergoes a rapid drop down to 14.04 $cm^3{\cdot}K{\cdot}mol^{-1}$ at 1.8 K indicating antiferromagnetic interactions in **3**. Based on the X-ray structure, the magnetic susceptibility data can be modeled as the sum of the susceptibility of the trinuclear clusters { $\chi_{Fe3O}(T)$ } with contributions from the seven isolated Fe^{III} ions with $S_{Fe} = 5/2$):

$$\chi_0(T) = \chi_{Fe3O}(T) + 7\left\{\frac{Ng^2\beta^2}{3kT} S_{Fe}(S_{Fe}+1)\right\}$$

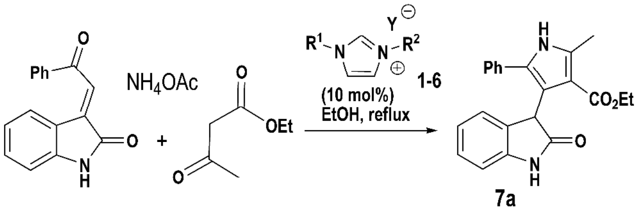
The following definition of the susceptibility, which accounts for contributions from the intermolecular interactions, has been used

$$\chi(T) = \frac{\chi_o(T)}{[1-(2zJ'\chi_o(T)/Ng^2\beta^2)]}$$

where χ_o is the susceptibility of the noninteracting paramagnetic centers in **3** (Fe_3O and 7 Fe^{III}), z is the number of nearest neighbors, and J' is the magnetic interactions between units.[10] The magnetic susceptibilities $\{\chi_{Fe3O}(T)\}$ of the spin frustrated $\{Fe^{III}{}_3O\}$ cluster were calculated using the Hamiltonian: $H=-2J_a(S_1S_2+S_2S_3)-2J_bS_1S_3$ where J_a and J_b are exchange parameters and $S_1 = S_2 = S_3 = 5/2$ the spin on the individual Fe^{III} ions. The eigenvalue of the spin-Hamiltonian was determined by using the Kambe vector coupling method.[11] Fits of the experimental data to the final expression of the susceptibility yielded one satisfactory set of parameters with $J_a = -22.8(6)\ cm^{-1}$, $J_b = -41.9(9)\ cm^{-1}$, g = 2.02(1) and $zJ' = -0.27(2)$ (Fig. S8). These values are in good agreement with other reported $\{Fe^{III}{}_3O\}$ cases.[9, 12, 13]

Mössbauer spectra of polycrystalline samples of **3** at T = 50 and 3 K are typical for paramagnetic behavior with an asymmetric quadrupole doublet which can be fitted using two doublets with isomer shifts (δ) 0.43 (0.43) and 0.32 (0.33) mm/s and quadrupole splittings (ΔE_Q) 0.60 (0.62) and 0.17 (0.19) mm/s, respectively for $\{Fe_3O\}^{7+}$ and $7[FeCl_4]^-$, typical of high spin S=5/2, Fe^{III} ions (details in Table S2, Figure S9).

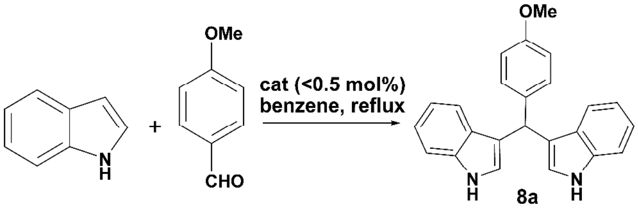
Pyrroles and oxindoles are important heterocyclic scaffolds of medicinal and therapeutic relevance.[14] Devising reactions to achieve one-step (concerted) multi-bond formation is a challenge in heterocycle and natural product synthesis. Recently, one-pot procedures have been developed for $InCl_3$ (20 mol%) catalyzing the synthesis of 2-methyl-4-(2-oxo-2,3-dihydro-1*H*-3-indolyl)-5-phenyl-1*H*-3-pyrrolecarboxylates.[15] This procedure does not require the isolation of the initial Michael addition product prior to the Paal-Knorr condensation. However, $InCl_3$ is harmful, corrosive and, moreover, expensive. Thus the development of eco-friendly alternatives, such as task-specific ionic liquids, would extend the scope of this approach. The new compounds **1, 2** and **4-6** can be used as promoters for two useful organic reactions. Compound **3** was excluded from this study since its thermal instability and technologically inefficient, time-consuming synthesis (crystallization takes about 2-3 month at 0°C) preclude useful applications. At the outset of the study, the synthesized compounds **1, 2** and **4-6** were tested (the results are summarized in Table S3) in the selected reaction of ammonium acetate, ethyl acetoacetate with 3-phenacylideneoxindole in EtOAc at 77°C for the synthesis of ethyl 2-methyl-4-(2-oxo-2,3-dihydro-1*H*-3-indolyl)-5-phenyl-1*H*-3-pyrrolecarboxylate (**7a**).



Scheme 1. Synthesis of 2-pyrrolo-3'-yloxindole (**7a**).

Compounds **4** and **5** gave the best results for these catalysts with product yields of 98 % (Table 1). By optimizing the reaction conditions, it was found that ~0.313 mol% of **1** was sufficient for the completion of the reaction with good yield. Another aim of our study was to investigate the recycling of catalysts. It is worth noting that the catalysts can be reused at least ten times without significant loss of activity for **4-6** (89÷92%) and a maximum of five times for **1** and **2** (65÷69%).

Among various indole derivatives, bis(indolyl)methanes are found in cruciferous plants and known to promote beneficial oestrogen metabolism and induce apoptosis in human cancer cells.[16] In order to study the scope of the properties of the compounds, these were also investigated as promoters for the condensation of indole with 4-methoxybenzaldehyde.[17] Refluxing of indole with 4-methoxybenzaldehyde in the presence of the synthesized ionic liquids as catalyst resulted in the selective formation of substance **8a** (Table S4). In all cases, the product was isolated by decantation from the catalyst followed by silica gel chromatography. Optimum results were obtained using ~0.315 mol % of **2** (Table S4). After a few cycles it was found necessary to increase the reaction time in order to achieve this optimum yield. The best overall yield (92%) was obtained using **2** in benzene (Table 1). In particular, all the catalysts apart from **5** are more effective than the known $FeCl_3$/[omim]PF_6 system which has an optimum yield of 78 %.[17]



Scheme 2. Synthesis of 3-((1H-indol-3-yl)(4-methoxyphenyl)methyl)-1H-indole (**8a**).

In summary, a series of ionic liquids containing antiferromagnetically coupled $\{Fe^{III}{}_3O(RCOO)_6L_3\}^{7+}$ cationic triangles has been synthesized and characterized by spectroscopic methods, magnetism and X-ray crystallography. It was demonstrated that this combination of metal ions (iron) with task-specific ionic liquid ligands can be used as new efficient catalysts for the successful high-yield synthesis of heterocyclic compounds within a green chemical protocol.

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Notes and references

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† Electronic Supplementary Information (ESI) available: Crystallographic data for **1**-**6** (CCDC no. 896157-896162) in CIF format. The synthetic protocols, thermal TG/DSC analysis and fragments of molecular structures. See DOI: 10.xxxx/b000000x/

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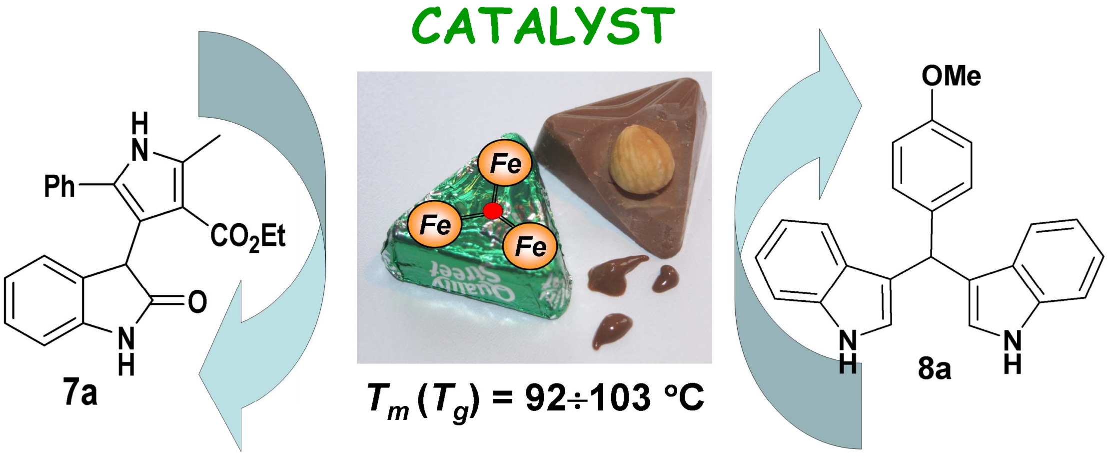
Table of Contents (TOC)

Catalytic “Triangles”: Binding of Iron in Task-Specific Ionic Liquids

Authors: Denis Prodius, Fliur Macaev, Eugenia Stingaci, Vsevolod Pogrebnoi, Valeriu Mereacre, Ghenadie Novitchi, George E. Kostakis, Christopher E. Anson, and Annie K. Powell



A new class of catalytically active task-specific ionic liquids with antiferromagnetically coupled $\{Fe^{III}{}_3O(RCOO)_6L_3\}^{7+}$ cations has been synthesized and structurally characterized.