

mer enhanced the efficiency of the energy or electron transfer reaction from the conjugated backbone to the metal complex.

### Experimental

**2:** A mixture of 2,5-dimethylbenzaldehyde (5.00 g, 37.3 mmol) and 2-acetylpyridine (4.51 g, 37.3 mmol) in 15 mL of methanol was cooled in ice, and then 10 mL of 3 wt.-% methanolic potassium hydroxide solution was added. After the mixture had been stirred overnight in ice, 1-(2-pyridylcarbonylmethyl)pyridinium iodide (10.83 g, 33.2 mmol) and ammonium acetate (26 g, 330 mmol) were added to this mixture and refluxed for 24 h. The residue resulting from evaporation was purified using column chromatography on activated alumina by eluting with  $\text{CHCl}_3$ . Recrystallization from methanol gave pale yellow crystals (yield 5.06 g, 57%).  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  8.46–8.68 (m, 6H, pyridine), 7.80–7.96 (m, 2H, pyridine), 7.25–7.40 (m, 2H, pyridine), 6.82–7.10 (m, 3H, phenyl-H), 2.30 (s, 6H,  $\text{CH}_3$ ). Anal. Calcd. for  $\text{C}_{23}\text{H}_{19}\text{N}_3$ : C 81.87, H 12.45, N 5.68. Found: C 81.83, H 12.41, N 5.72.

**3:** Benzoyl peroxide (0.03 g, 0.12 mmol) was added to a suspension of **2** (1.00 g, 2.96 mmol) and *N*-bromosuccinimide (NBS) (1.06 g, 5.93 mmol) in 30 mL of  $\text{CCl}_4$ , and refluxed for 3 h. The residue resulting from evaporation was washed with water and dried. The precipitates were filtered and dissolved in  $\text{CHCl}_3$ . Reprecipitation in methanol gave 2,5-bis(bromomethylphenyl)-2,2':6',2''-terpyridine as a white solid (yield 4.08 g, 92%).  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  8.45–8.70 (m, 6H, pyridine), 7.80–7.95 (m, 2H, pyridine), 7.1–7.42 (m, 5H, pyridine and phenyl-H), 4.58 (s, 4H,  $\text{CH}_2\text{Br}$ ). A mixture of 2,5-bis(bromomethylphenyl)-2,2':6',2''-terpyridine (2.00 g, 4.04 mmol) and triphenyl phosphine (2.23 g, 8.49 mmol) in 20 mL of ethanol was stirred at room temperature for 24 h. The residue resulting from evaporation was washed with acetone several times. It weighed 2.42 g (yield 59%) after drying under vacuum.  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  8.70–9.20 (m, 6H, pyridine), 8.10–8.45 (m, 2H, pyridine), 7.40–7.88 (m, 35H, pyridine and phenyl-H), 4.88 (d, 4H,  $\text{CH}_2\text{P}(\text{Ph})_3$ ).

**1:** 1.6 M of *n*-butyllithium in hexane was added to a solution of **3** (0.51 g, 0.5 mmol) and 2,5-bis(hexyloxy)benzene-1,4-dialdehyde (0.167 g, 0.5 mmol) in dry THF, and the reaction mixture was stirred for 10 h at room temperature. The mixture was poured into methanol to precipitate the polymer. Purification was accomplished by two reprecipitations into methanol. Removal of solvent under vacuum afforded polymer **1** as an orange solid with an average molecular weight of 4000 based on polystyrene standard by GPC (0.22 g, 70%). The composition of the copolymer was determined by elemental analysis of carbon and nitrogen.  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  8.80 (br, pyridine), 7.87–7.94 (br, pyridine), 7.49–7.8 (m, vinyl-H), 7.26–7.40 (br, pyridine), 6.89 (br, phenyl-H), 4.09 (br,  $\text{OCH}_2(\text{CH}_2)_4\text{CH}_3$ ), 1.90 (br,  $\text{OCH}_2\text{CH}_2(\text{CH}_2)_3\text{CH}_3$ ), 1.53 (br,  $\text{OCH}_2\text{CH}_2\text{CH}_2(\text{CH}_2)_2\text{CH}_3$ ), 1.26 (br,  $\text{OCH}_2\text{CH}_2\text{CH}_2(\text{CH}_2)_2\text{CH}_3$ ), 0.85 (br,  $\text{OCH}_2\text{CH}_2\text{CH}_2(\text{CH}_2)_2\text{CH}_3$ ). Anal. Calcd. for  $(\text{C}_{23}\text{H}_{15}\text{N}_3)_{0.51}(\text{C}_{20}\text{H}_{30}\text{O}_2)_{0.49}$ : C 81.20, H 7.19, N 6.48. Found: C 81.74, H 6.87, N 6.65.

Received: August 11, 1997  
Final version: November 24, 1997

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### Oxidized Thin Films of $\text{C}_{60}$ : A New Humidity-Sensing Material\*\*

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Since the discovery and isolation of  $\text{C}_{60}$ , its conductive properties have been the subject of much scrutiny. In the ground state,  $\text{C}_{60}$  has been found to be an insulator with photoconductivity strongly influenced by the presence of sorbed oxygen.<sup>[1]</sup> The intercalation of alkali metals into  $\text{C}_{60}$  has yielded—depending on type and stoichiometry of alkali metal used—phases with electronic conductivity ranging from insulating to metallic.<sup>[2–5]</sup> These alkali phases have been shown to be unstable with respect to oxidation in air.<sup>[6,7]</sup> Unseen in the literature, however, is any form of pure or derivatized  $\text{C}_{60}$  showing significant ionic conductivity. Further, there are still relatively few examples of fullerene materials with obvious application potential. We report here the discovery of a material derived from alkali metal-doped  $\text{C}_{60}$  that displays a conductivity that is mediated by the amount of ambient water vapor to which it is exposed, and whose response is quick enough to easily follow a human's breathing from a distance of 30 cm. The AC admittance of the material as a film has been measured over a broad range of water vapor pressure, and is seen to change by nearly three orders of magnitude on going from less than 0.03 mbar  $\text{H}_2\text{O}$  to 25 mbar  $\text{H}_2\text{O}$ . Although UV-vis spectra of the film material show minimal deviations from the absorption spectra of pure  $\text{C}_{60}$ , the near-IR photoacoustic response of the bulk material indicates the presence of highly functionalized species. DC polarization of the material suggests a notable ionic contribution to the conductivity, which may be due to proton conduction or an electrochemical reaction.

The first films of this material were the product of the oxidation in air of previously described films of  $\text{K}_3\text{C}_{60}$ ,<sup>[8]</sup>

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[\*\*] We thank Anne Davidson for collection of the bulk photoacoustic spectrum. This work was supported by the Office of Naval Research under N00014-96-1-0053, and the National Science Foundation under DMR 95-20971.