Table I. 5′-^{32}P-GAGC$_4$GAGC$_8$G(C$_8$, 1′-^{1}H) or 5′-^{32}P-GAGC$_4$GAGC*G(C$_8$*, 1′-^{2}H) and (CGCT)$_3$ with NCS Quantitation of Isotope Effects Using Densitometry

	C$_8$: 1′-^{1}H		C$_8$*: 1-^{2}H			
experiment	area C$_4$[a]	area C$_8$[a]	area C$_4$[a]	area C$_8$*[a]	$\frac{C_8[1'\text{-}^1H]}{C_8^*[1'\text{-}^1H]}$	$\frac{C_4[1'\text{-}^1H]}{C_4[1'\text{-}^2H]}$
NCS (20 μM + alkali)	2.8	1.4	2.7	0.4	3.3	1.0
NCS (40 μM + alkali)	4.1	2.0	4.0	0.5	4.2	1.0

[a] Areas under the peaks were established by using an LKB Ultroscan XL Laser densitometer. The areas reported were obtained after subtraction of a small amount of background observed in control experiments run in the absence of drug and in the presence of alkali (see for example, lane 2, Figure 2). Typical backgrounds are less than 10% of the reported areas. C$_4$ serves as the internal control as both oligomers contain C$_4$ with ^{1}H at C-1′.

method for the detection of the chemistry of minor lesions.

[1′-^{2}H]dC*TP and [1′-^{1}H]dCTP[6] were incorporated specifically into the penultimate residue of the nanomer d(GAGC$_4$GAGC$_8$ (or C$_8$*)G) as previously described.[4] The nanomer containing unlabeled (C$_4$) and (C$_8$) in the d(AGC) sequence was end labeled, annealed to d(CGCT)$_3$, and incubated with limiting concentrations of NCS, and the degradation products were analyzed by polyacrylamide gel electrophoresis. In Figure 2 it can be seen that there is alkali dependent cleavage at both C$_8$ and C$_4$ (compare lanes 5, 7. and 6, 8). Identical experiments were performed on the same oligomer containing deuteriated C$_8$ (C$_8$*). The direct observation of an isotope effect on NCS mediated oligomer damage is apparent on the alkali labile reaction by comparison of lanes 7 with 11 and 8 with 12. Quantitation of the isotope effect by densitometry is summarized in Table I. A k_H/k_D of 3.3 and 4.2 for 1′-C-H bond cleavage was calculated at 20 and 40 μM NCS, respectively. A similar analysis for cleavage at C$_4$, the internal control, gave the expected k_H/k_D of 1.0. The non-"alkali" dependent cleavage observed in lanes 5 and 6 with migration slightly faster than G$_7$ is presumably an intermediate d(GAG) moiety attached to a 5′-phosphorylated α,β-unsaturated lactone, that is, the precursor to C$_4$. Since the 1′-hydrogen of C$_4$ is unlabeled, there is no isotope effect on its formation (compare lanes 5, 6 with lanes 9, 10). These results provide the first direct evidence that NCS can effect removal of a 1′-hydrogen from a deoxycytidine residue in the d(AGC) sequence and demonstrate that a substantial isotope effect occurs on this reaction.

When the protio and deuterio nanomers were annealed to d-(CICT)$_3$ instead of d(CGCT)$_3$, no significant isotope effect was observed (data not shown). Previous studies have shown that inosine (I), base-paired to C, enhances NCS-mediated cleavage 5-fold relative to G. If this enhancement is due to an increase in the relative rates of hydrogen abstraction by the activated drug versus dissociation from DNA of the activated drug, then the suppression of the isotope effect is explainable. A similar modulation of isotope effects was observed with BLM.[1]

These studies have recently been extended to an EcoRI-BamHI DNA fragment from pBR322 (375 bp) in which [1′-^{2}H]dC or [1′-′H]dC has been incorporated.[1] Two d(AGC) sequences are present in this restriction fragment:

^{32}P–GAC$\underline{\text{AGC}}$TTATCATCGATA$\underline{\text{AGC}}$T–

Results from densitometry scans (data not shown) indicated an alkaline labile isotope effect of 3.7 on the cleavage of dC closest to the 5′-end. No detectable damage was observed at the second d(AGC) sequence. The extent of damage at dC in the first d(AGC) sequence was less than 0.1% of the total damage. Such pathways, while difficult to investigate from a mechanistic point of view, may be the pathway of major biological significance. The direct observation of this C-1′ C–H bond cleavage thus demonstrates the power of this method in establishing the chemistry involved in minor pathways.

Acknowledgment. This research was supported by the National Institutes of Health GM 34454 (J.S. and J.W.K.) and National Institutes of Health CA 44257 and GM 12573 (I.H.G.).

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Unusual Molecular Hydrogen Complex of Rhenium: A Long Hydrogen–Hydrogen Bond and Inertness to Substitution

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Since the discovery of M(H$_2$)(CO)$_3$(PR$_3$)$_2$ (M = Mo, W; R = *i*-Pr or Cy) by Kubas and co-workers,[1] transition-metal polyhydrido complexes have been the subject of intensive research and close scrutiny with respect to the nature of metal–hydrogen bonding,[2] e.g., M–(H)$_3$ (classical), H–M–(H$_2$) (nonclassical), or possibly M–(H$_3$). In this context, the question of the bonding in the rhenium pentahydrides ReH$_5$L$_3$ has been particularly intriguing, since initially ^{1}H NMR (T_1 values) and X-ray results pointed to different conclusions when L = PPh$_3$.[3] Here we report that protonation of an analogous rhenium pentahydride containing a tridentate phosphine ligand, viz., ReH$_5$(Cyttp) (**1**, Cyttp = PhP[CH$_2$CH$_2$CH$_2$PCy$_2$]$_2$), affords an unusual dihydrogen complex, [Re(H$_2$)H$_4$(Cyttp)]SbF$_6$ (**2**). This product features a surprisingly long H–H bond; it also exhibits a remarkable lack of reactivity in substitution reactions with D$_2$ and typical 2e donor ligands.

1 was prepared by the action of excess NaBH$_4$ on ReCl$_3$(Cyttp) in ethanol and characterized as a classical pentahydride by ^{1}H NMR spectroscopy (T_1) and X-ray crystallography.[4] It reacts with excess HSbF$_6$ (65% aqueous) in benzene solution to afford **2**[5-7] as an air-stable white solid in high (>88%) yield. **2** is remarkably resistant to loss of H$_2$; e.g., it remains intact after storage at 0.1 Torr for more than 2 days at ambient temperatures.

Variable-temperature ^{1}H NMR spectra of **2** at 250 MHz in CD$_2$Cl$_2$ solution are shown in Figure 1. At ambient temperature,

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[†] Deceased December 7, 1988.

(1) (a) Kubas, G. J.; Ryan, R. R.; Swanson, B. I.; Vergamini, P. J.; Wasserman, H. J. *J. Am. Chem. Soc.* **1984**, *106*, 451. (b) Wasserman, H. J.; Kubas, G. J.; Ryan, R. R. *J. Am. Chem. Soc.* **1986**, *108*, 2294. (c) Kubas, G. J.; Ryan, R. R.; Wrobleski, D. *J. Am. Chem. Soc.* **1986**, *108*, 1339. (d) Kubas, G. J.; Unkefer, C. J.; Swanson, B. I.; Fukishima, E. *J. Am. Chem. Soc.* **1986**, *108*, 7000.

(2) Reviews: (a) Kubas, G. J. *Acc. Chem. Res.* **1988**, *21*, 120. (b) Crabtree, R. H.; Hamilton, D. G. *Adv. Organomet. Chem.* **1988**, *28*, 299.

(3) (a) Hamilton, D. G.; Crabtree, R. H. *J. Am. Chem. Soc.* **1988**, *110*, 4126. (b) Cotton, F. A.; Luck, R. L. *Inorg. Chem.* **1989**, *28*, 6. (c) Cotton, F. A.; Luck, R. L. *J. Am. Chem. Soc.* **1989**, *111*, 5757. (d) Luo, X.-L.; Crabtree, R. H. *Inorg. Chem.*, in press.

(4) Kim, Y.; Meek, D. W. Abstracts, 21st Central Regional Meeting of the American Chemical Society, Cleveland, Ohio, May 31–June 2, 1989; No. 233. Spectroscopic and crystallographic data are provided in supplementary material; they will be published in a full paper with J. Gallucci.

(5) **2**: ^{31}P{^{1}H} NMR (C$_6$D$_6$): 12.49 (d), –8.06 ppm (t) (J_{PP} = 23.7 Hz). Anal. Calcd for C$_{36}$H$_{67}$F$_6$P$_3$ReSb: C, 42.61; H, 6.65. Found: C, 42.73; H, 6.68.

(6) Similar protonation reactions of ReH$_5$L$_3$ (L = PPh$_3$,[7a] PMe$_2$Ph[7b]) are reported to give [ReH$_6$L$_3$]$^+$; [ReH$_6$(PPh$_3$)$_3$]BF$_4$ was formulated as a nonclassical, η^2-H$_2$-containing complex from electrochemical evidence.[7c]

(7) (a) Moehring, G. A.; Walton, R. A. *J. Chem. Soc., Dalton Trans.* **1987**, 715. (b) Douglas, P. G.; Shaw, B. L. *Inorg. Synth.* **1977**, *17*, 64. (c) Costello, M. T.; Walton, R. A. *Inorg. Chem.* **1988**, *27*, 2563.

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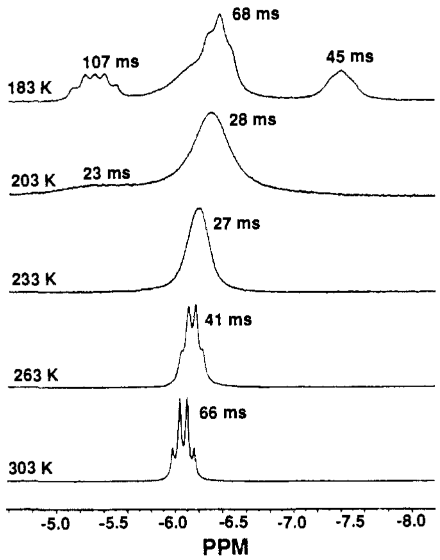

Figure 1. Variable-temperature ^1H NMR spectra (250 MHz, CD_2Cl_2) of **2** in the metal-hydride region. The T_1 values are written on the peaks.

the hydride resonance appears as a quartet at −6.06 ppm with J_{PH} = 15.3 Hz. Upon cooling to 183 K, three broad peaks are observed with relative intensities 1:4:1. The signals at −5.31 and −7.39 ppm are assigned to terminal hydrogens on the basis of their unit intensities. The overlapping peaks centered at −6.36 ppm derive from the presence of four remaining hydrogens. The T_1 values decrease as the temperature is lowered from 303 K, reach a minimum at 233 K, and then increase. The minimum T_1 value of 27 ms at −40 °C strongly implicates the presence of η^2-H_2 by the criteria developed by Crabtree[3a,8] and by their subsequent slight modifications.[3b-d,9,10]

A low-temperature (−50 °C) X-ray diffraction analysis supports the foregoing conclusion by revealing the location of a molecular hydrogen ligand.[11] The overall geometry about the rhenium atom (Figure 2) is that of a distorted triangulated dodecahedron (Re is 0.08 Å out of the plane of P(1), H(1), and H(2)) and closely resembles that of **1** by replacement of the hydride trans to the central phosphorus by η^2-H_2. The H–H bond distance of 1.08 (5) Å[12,13] is noticeably longer than those found in the three η^2-H_2 metal complexes studied by X-ray crystallography (0.75 (16),[1a,12] 0.89 (11),[15] and 0.80 (6) Å[16]).[17,18] Interestingly, it is comparable with the H–H bond distance of 1.06 Å calculated for the one-electron H_2^+ ion.[19] The present X-ray structure is the first of a molecular hydrogen complex in which the metal adopts a coordination number greater than six and which is not of the d^6 configuration.

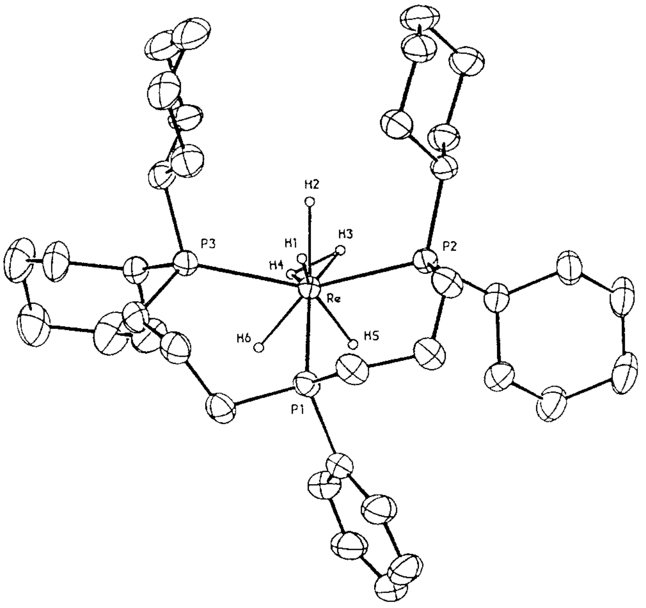

Figure 2. ORTEP plot of **2**. Non-hydrogen atoms are represented by 50% probability thermal ellipsoids. Hydrogen atoms bonded to Re are drawn with an arbitrary radius; other hydrogen atoms are omitted for clarity.

The unusually long H–H bond distance of the η^2-H_2 in **2** may be responsible for the remarkable lack of reactivity of this complex to substitution.[20] Accordingly, no reaction is observed when **2** is treated with each of CO, N_2, *t*-BuNC, and $P(OMe)_3$ in benzene at room temperature for 12 h, or with D_2 at ambient temperatures even under UV irradiation (Hanovia 450-W medium-pressure Hg lamp) for at least 2 h. However, addition of a base, e.g., CHO_2Na, $MeCO_2Na$, PMe_3, or NEt_3, to **2** in acetone leads to a quantitative regeneration of **1** within 30 min at ca. 25 °C.

The aforementioned structural data and the lack of reactivity of **2** to substitution strongly suggest that the Re–(H_2) interaction in this complex represents a substantial progression along the reaction coordinate from M–(H_2) to M–$(H)_2$. Previous work by Kubas and co-workers has established the occurrence of an equilibrium between $W(H_2)(CO)_3[P(i\text{-}Pr)_3]_2$ and $WH_2(CO)_3[P(i\text{-}Pr)_3]_2$ in solution;[1c] similar equilibria exist for $[CpRu(dmpe)(H_2)]^+$ (dmpe = $Me_2PCH_2CH_2PMe_2$) and $[Re(H_2)H_2(CO)(PMe_2Ph)_3]BF_4$.[21,22] These earlier observations have suggested that there may be two distinct classes of hydride, rather than a smooth gradation of structure.[2b] Our results show that the latter behavior may occur as well.

Acknowledgment. We are grateful to the National Science Foundation (Grant CHE-8420806 to A.W.) and The Ohio State University for financial support and to Professors K. G. Caulton, R. H. Crabtree, and R. H. Morris and Dr. G. J. Kubas for helpful

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(8) Classical metal hydrides were noted to show T_1(min) values >150 ms, and nonclassical hydrides, <80 ms, at 250 MHz.[3a]

(9) It was suggested[3b-d,10] that the previously proposed[3a] upper limit of the T_1 values used to predict nonclassical hydrides is too high.

(10) Cotton, F. A.; Luck, R. L. *Inorg. Chem.* **1989**, *28*, 2181.

(11) Crystal data for $[Re(H_2)H_4(Cyttp)]SbF_6$: space group $P\bar{1}$ with a = 9.874 (2) Å, b = 14.242 (4) Å, c = 16.198 (2) Å, α = 99.12 (2)°, β = 98.85 (2)°, γ = 109.42 (2)°, V = 2068.3 Å^3, Z = 2, D_{calcd} = 1.629 g cm^{-3}; data collected at −50 °C with Mo Kα radiation, μ = 37.9 cm^{-1}, 2θ limits 4–55°, 449 variables refined with 7982 unique reflections with $F_o^2 > 3\sigma(F_o^2)$ to $R(F)$ = 0.022 and $R_w(F)$ = 0.032, GOF = 1.45. The structure was solved by the combination of direct methods (MULTAN 11/82) and Fourier synthesis. The six hydrogen atoms bonded to Re were located on a difference Fourier map and refined isotropically. Selected bond distances and interatomic separations: Re–H(1) 1.63 (3), Re–H(2) 1.68 (3), Re–H(3) 1.55 (4), Re–H(4) 1.45 (3), Re–H(5) 1.73 (4), Re–H(6) 1.68 (3), H(3)–H(4) 1.08 (5) Å; shortest H···H, H(2)···H(3) 1.61 (5) Å.

(12) Neutron diffraction is more reliable than X-ray diffraction for the determination of H–H bond distances, provided there is no rotational disorder of H_2; for comparison, H–H length in $W(H_2)(CO)_3[P(i\text{-}Pr)_3]_2$ is 0.75 (16) Å (X-ray) and 0.84 Å (neutron).[1a]

(13) Significantly, when this bond distance is used and slow rotation of H_2 is assumed, a T_1 value of 25 ms is calculated,[3a,14] in good agreement with the experimental finding of 27 ms.

(14) Bautista, M. T.; Earl, K. A.; Maltby, P. A.; Morris, R. H.; Schweitzer, C. T.; Sella, A. *J. Am. Chem. Soc.* **1988**, *110*, 7031.

(15) For $[Fe(H_2)H(dppe)_2]BF_4$ (dppe = $Ph_2PCH_2CH_2PPh_2$); see: Morris, R. H.; Sawyer, J. F.; Shiralian, M.; Zubkowski, J. D. *J. Am. Chem. Soc.* **1985**, *107*, 5581. For neutron diffraction of the BPh_4^- salt, see: Ricci, J. S.; Koetzle, T. F.; Bautista, M. T.; Hofstede, T. M.; Morris, R. H.; Sawyer, J. F. *J. Am. Chem. Soc.* **1989**, *111*, 8823.

(16) For $(P\text{-}N)(H_2)Ru(\mu\text{-}Cl)_2(\mu\text{-}H)RuH(PPh_3)_2$ (P-N = $Fe[\eta^5\text{-}C_5H_3\text{-}(CHMeNMe_2)P(i\text{-}Pr)_2\text{-}1,2](Cp)$; see: Hampton, C.; Cullen, W. R.; James, B. R.; Charland, J. P. *J. Am. Chem. Soc.* **1988**, *110*, 6918.

(17) In addition, neutron diffraction of $Fe(H_2)H_2(PEtPh_2)_3$ showed a H–H distance of 0.821 (10) Å; see: Van Der Sluys, L. S.; Eckert, J.; Eisenstein, O.; Hall, J. H.; Huffman, J. C.; Jackson, S. A.; Koetzle, T. F.; Kubas, G. J.; Vergamini, P. J.; Caulton, K. G. *J. Am. Chem. Soc.*, in press.

(18) However, the H–H bond distance in $[Os(H_2)H(depe)_2]BF_4$ (depe = $Et_2PCH_2CH_2PEt_2$) calculated from T_1 measurements is 1.12 ± 0.03 Å (rapid rotation of H_2) or 1.40 Å (slow rotation of H_2).[14]

(19) Schaad, J. L.; Hicks, W. V. *J. Chem. Phys.* **1970**, *53*, 851.

(20) For chemical behavior of molecular hydrogen complexes, see ref 2b.

(21) Another example is $[Re(H_2)H_2(CO)(PMe_2Ph)_3]BF_4$: Luo, X.-L.; Crabtree, R. H. *J. Chem. Soc., Chem. Commun.*, in press.

(22) Chinn, M. S.; Heineky, D. M. *J. Am. Chem. Soc.* **1987**, *109*, 5865.

comments and for providing us with preprints of their work.

Supplementary Material Available: Spectroscopic data and details of structure determination of **1**, including ORTEP, crystal data, data collection and refinement, positional parameters, temperature factor expressions, and selected bond distances and angles, and tables of positional parameters, temperature factor expressions, and selected bond distances and angles for **2** (32 pages). Ordering information is given on any current masthead page.

Synthesis of Self-Doped Conducting Polyaniline

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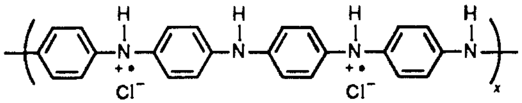
Polyaniline was the first conducting polymer whose electronic properties could be reversibly controlled by both protonation and charge-transfer doping.[1,2] We report the synthesis of sulfonic acid ring-substituted polyaniline, the first protonic acid self-doped conducting polymer. Without external doping, the polymer has a conductivity of $\sim$0.1 S/cm, which is independent of pH when equilibrated with aqueous acid solutions of pH less than or equal to 7.

Emeraldine hydrochloride powder (I) was synthesized from an aqueous solution of aniline, $(NH_4)S_2O_8$, and HCl. It was then converted to analytically pure emeraldine base (II) via a previously described method.[3] Emeraldine base (0.5 g) was sulfonated by being dissolved in 40 mL of fuming sulfuric acid with constant stirring. The color of the solution changed from dark purple to dark blue during $\sim$2 h at room temperature. The solution was then slowly added during $\sim$20 min to 200 mL of methanol to precipitate most of the product, the temperature being held between 10 and 20 °C by an ice bath. Precipitation was completed by the addition of 100 mL of acetone. The green powder was then collected on a Büchner funnel, and the precipitate cake was washed at least 10 times with $\sim$50-mL portions of methanol until the filtrate had a pH of 7 when tested by wet pH paper. The liquid level in the Büchner funnel was constantly adjusted so that it remained above the top of the precipitate in order to prevent cracking of the precipitate cake, which would result in inefficient washing. It was then permitted to remain under suction for approximately 10 min; the resulting precipitate cake was slightly soluble in water, giving a green solution. The filter cake then was transferred on the filter paper to a vacuum desiccator and dried under dynamic vacuum for 24 h. Elemental analyses[4] are consistent with structures given by compound IIIA or by compound IIIB containing two molecules of water of hydration. Either structure would give the polysemiquinone radical cationic (polaronic) structure, and both are consistent with presently available experimental data for a ring-sulfonated, protonated form of polyaniline in the emeraldine oxidation state.[5] The polymer is very soluble in dilute aqueous base, giving a blue-violet solution of the corresponding undoped (insulating) salt form, compound IV, or the alternate structure having the SO_3^- groups on adjacent rings.



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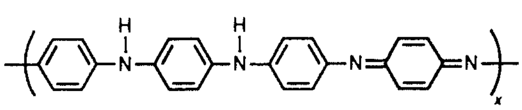

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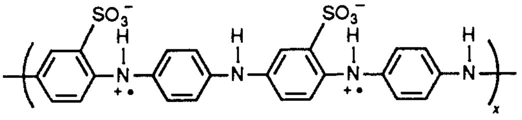

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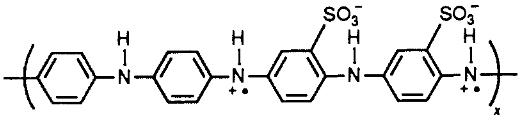

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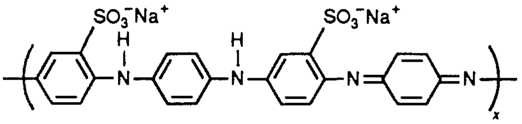
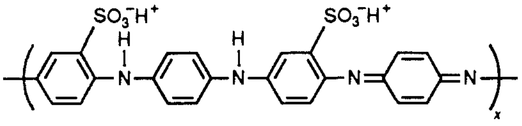


IV

The sodium salt was synthesized by dissolving 0.5 g of compound IIIA,B in 20 mL of 0.1 M NaOH solution. Water was removed from the solution by slowly evacuating the vessel at room temperature. After most of the water had been removed, the product was transferred to a vacuum desiccator and dried under dynamic vacuum for 24 h. The polymer was obtained as a free-flowing dark purple powder.

The elemental analysis, infrared and electron spin resonance spectra, and conductivity are consistent with compound IIIA,B with UV-vis polaron band transitions[6] at 435 nm (2.85 eV) and 850 nm (1.46 eV), a π–π^* transition[7] at 320 nm (3.88 eV), and an intense ESR signal of line width $\sim$0.4 G. This self-doped polymer can be regarded as being formed hypothetically via the initial formation of the strong acid, compound V, which then immediately protonates ("dopes") the imine nitrogen atoms to give the conducting polymer in an analogous manner to the protonation of the parent emeraldine base polymer by HCl.[8] Treatment of compound IIIA,B with aqueous NaOH yields the corresponding nonprotonated sodium salt, compound IV, an insulator, analogous to emeraldine base (II).



V

It is implicit from the structure of compound IIIA,B that, under the experimental conditions employed, only half the rings are sulfonated. This is consistent with the fact that only half the rings

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[1] (a) MacDiarmid, A. G.; Chiang, J. C.; Halpern, M.; Huang, W. S.; Mu, S. L.; Somasiri, N. L. D.; Wu, W.; Yaniger, S. I. *Mol. Cryst. Liq. Cryst.* **1985**, *121*, 173-180. (b) Epstein, A. J.; MacDiarmid, A. G. *Proc. Int. Winter School On Electronic Polymer Properties*; Kirchberg, Austria; March 1989; *Solid State Science* **1989**, *91*, 282-289; Kuzmany, H., Ed.; Springer-Verlag: Berlin. (c) MacDiarmid, A. G.; Epstein, A. J. *Trans. Faraday Soc.*, in press.

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[4] Elemental anal. Found for compound IIIA or compound IIIB: C, 52.12; H, 3.98; N, 10.13; S, 11.18; O, 22.59. (Oxygen by difference.) Calcd for $C_{12}H_9N_2SO_3(H_2O)_{0.98}$: C, 51.67; H, 3.96; N, 10.04; S, 11.49; O, 22.83.

[5] Copolymerization of 2-aminobenzenesulfonic acid and aniline yields a similar product. However, attempts at polymerization of 2-aminobenzenesulfonic acid alone were unsuccessful.

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