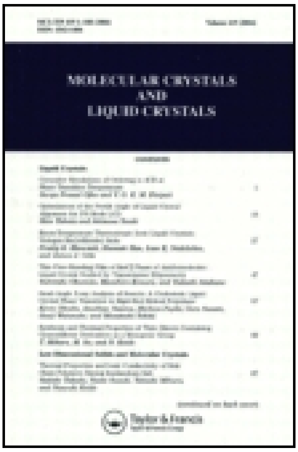

Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl19

New Organic Polymers And Molecules With Very High Spin States

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To cite this article: Dennis A. Dougherty , S. Joshua Jacobs , Scott K. Silverman , Michael M. Murray , David A. Shultz , Anthony P. West Jr & Jeffrey A. Clites (1993): New Organic Polymers And Molecules With Very High Spin States, Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals, 232:1, 289-304

To link to this article: http://dx.doi.org/10.1080/10587259308035719

Mol. Cryst. Liq. Cryst. 1993, Vol. 232, pp. 289–304
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Printed in the United States of America

NEW ORGANIC POLYMERS AND MOLECULES WITH VERY HIGH SPIN STATES

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Abstract: A number of new organic tetraradicals with quintet ground states have been prepared. Along with their intrinsic significance, these structures provide valuable tests of certain design principles for the construction of organic magnetic materials. The tetraradicals all follow the "bis(TMM)" approach, which promises to provide a wealth of information on spin preferences in organic structures. Efforts to prepare prototypes of the polaronic ferromagnet are also described. Current work is aimed at evaluating the role of bipolaron formation in such systems, in an effort to increase the absolute spin concentration.

INTRODUCTION

The immediate goal of our research in organic magnetism is to develop rational strategies for controlling the spin-spin interactions among radicals and radical ions. To that end we have been pursuing[1] the development of so-called ferromagnetic coupling units (Figure 1). These are simple building blocks that ensure a high spin coupling between two spin-containing structures, regardless of the nature of the spin-containing unit. We hasten to add that we are not alone in this pursuit, and that several other groups are making important advances along the lines of Figure 1.

In the present overview, we will emphasize two aspects of our current research program. The first is the "bis(TMM)" approach – a new and quite general strategy for evaluating potential ferromagnetic coupling units. Second, we will discuss our efforts to develop the polaronic ferromagnet, an especially interesting approach to polymer-based magnets.

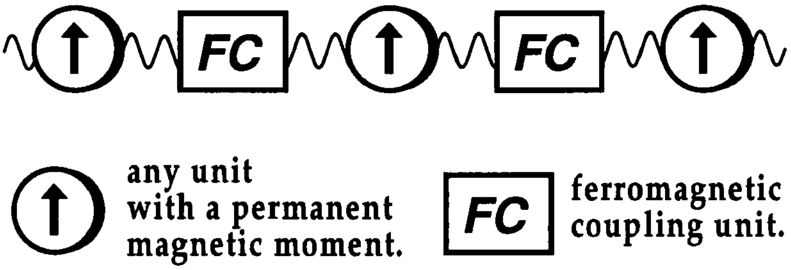


FIGURE 1 Schematic design for new magnetic materials.

THE BIS(TMM) APPROACH

Design

The design approach of Figure 1 requires robust ferromagnetic coupling units that will function with a wide range of spin containing units. From the many studies of triplet biradicals over the past two decades, a variety of candidates for ferromagnetic coupling units have emerged. However, new ferromagnetic coupling units would be very valuable, especially if they provide new capabilities relative to existing systems. Desirable attributes would include: the opening up of new synthetic routes to magnetic materials; an energetically stronger preference for the high spin state, which presumably would allow higher critical temperatures; a tolerance of substituents and chemical reactions or processing methods that may be necessary; and the ability to enforce ferromagnetic coupling in three dimensions.

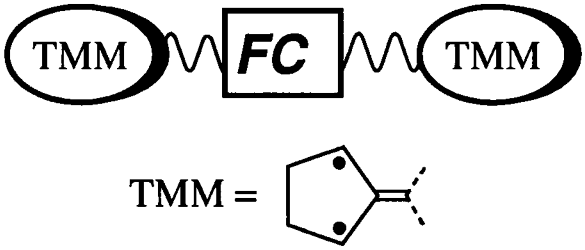


FIGURE 2 Schematic of the "bis(TMM)" approach.

We have recently been pursuing[2] a potentially general strategy for evaluating ferromagnetic coupling units that uses the design of Figure 2. The TMM is the trimethylenemethane derivative developed by Berson, in which the biradical is incorporated into a five-membered ring.[3] Conceptually, the bis(TMM) strategy is not unique. In essence it is just the polycarbene approach that has been so brilliantly exploited by Itoh, Iwamura, and others.[4] We do feel, however, that there are several important technical advantages to the bis(TMM) approach, that make it very much worth pursuing. These include:

1. The TMM unit is a "robust" triplet. The S-T gap is approximately 14 kcal/mol, and it remains large even with substantial substitution and/or structural modification.
2. With the Berson modification, the TMM is remarkably stable thermally. We have observed such triplets to be stable at least to room temperature, in rigid media.
3. The synthetic route to the TMMs' photochemical precursors – the bicyclic diazenes – is quite general and allows incorporation of the biradical into a wide range of structures.
4. The photochemistry of the diazenes and of the TMMs themselves is straightforward, efficient, and generally devoid of deleterious side reactions.
5. The EPR spectroscopy of the TMM unit is extremely well understood. As illustrated below, this can be of considerable value in assigning quintet EPR spectra, which are often only qualitatively informative at best.

Our initial applications of this strategy have been quite successful, and we will summarize them and some preliminary new results below. We believe we will be able to learn a great deal about design principles for magnets from this approach.

Initial Targets - EPR Results

Our first bis(TMM) structures are shown in Figure 3. They are meant to evaluate different ferromagnetic coupling units that have been suggested based on biradical studies. Of course, structure **1** uses m-phenylene, the longest known and most studied ferromagnetic coupling unit. In the present context, **1** is essentially a control molecule – there was little doubt at the outset that it would have a quintet ground state. Tetraradicals **2-4** test localized structures as ferromagnetic coupling units. For both cyclobutanediyl (**5**) and cyclopentanediyl (**6**) the parent biradicals and several derivatives have been shown to have triplet ground states.[5] This suggests they may serve as general ferromagnetic coupling units. Essentially nothing is known of the analogous cyclohexanediyl, and tetrayl **4** is meant to test whether it too can serve as a ferromagnetic coupling unit. Using an adamantyl system to deliver the six-member ring offers two advantages. It locks the ring into a chair conformation, thereby eliminating ambiguities associated with possible conformational issues. Also, if the six ring is a ferromagnetic coupling unit, an adamantane could become an example of the much sought three dimensional ferromagnetic coupling unit, with the ability to couple as many as six spin containing units. An additional, but by no means minor, point in the favor of adamantane is that the synthesis of the required bisdiazene was much easier in this system than for an analogous cyclohexane.

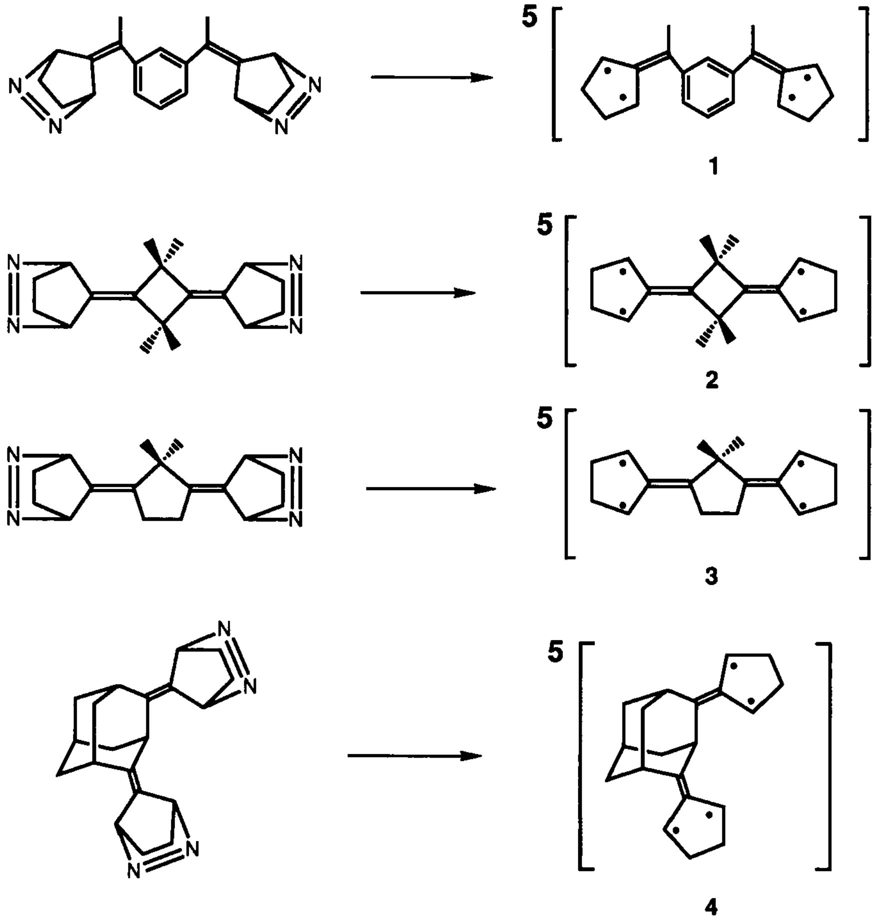
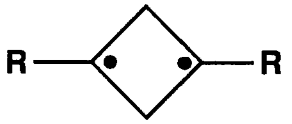


FIGURE 3 Initial tetraradical targets and bisdiazene precursors.

Before proceeding, we pause to note the pioneering contributions to this area by Gerhard Closs, who was the first to directly observe a localized biradical (**6**). This is one of many areas in which Closs made seminal contributions, and we note with great personal sadness his untimely passing.



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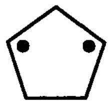

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With varying degrees of difficulty, the bisdiazene precursors to **1-4** (Figure 3) were all made. Photolysis at 77 K or lower was monitored by EPR. For **1**, **2**, and **3** a similar sequence of events ensued. At short photolysis times, the EPR signal was that of a simple TMM, reflecting loss of a single N_2 from the bisdiazene. Here, the well-established spectroscopy of the TMM unit was especially valuable, as the spectral assignments could be made with absolute certainty.

Upon extended photolysis the spectra underwent changes until, typically after 5 - 10 hours, no further change was seen. These new spectra were assigned to the quintet states of tetraradicals **1**, **2**, and **3**. Quintet, powder EPR spectroscopy is not a common technique, and is not as informative as single crystal spectroscopy of the sort that was most common in the initial polycarbene work. However, we have found that the EPR zero-field splitting (zfs) parameter D, which is most influential in determining the spectral features, can be predicted, *a priori.* Some time ago, Itoh, in the context of the polycarbene work, gave simple formulas for predicting zfs values for tetraradicals built up from two triplets interacting through a coupling unit.[6] It requires only an appropriately scaled addition of the zfs tensors for the three components: the two triplet building blocks and the coupling unit. Again, the very well understood spectroscopy of the TMM unit is valuable, and for **1-3**, the zfs parameters of the coupling units are also well characterized. Thus, it was a straightforward matter to predict the spectra for such species, and we find excellent agreement between theory and experiment, with no adjustable parameters.

Our assignments are further bolstered by full spectral simulations of the quintet spectra. We have written a computer program to perform such simulations, based on known procedures, and we find excellent agreement between simulated and observed spectra.

For tetraradicals **1** and **2**, the EPR spectra undergo no changes over a temperature range from 4 to 77 K, and the signal intensity follows the Curie Law. This suggests a quintet ground state, with a substantial gap to the next highest state. This is not, however, the case with **3**. Warming from 4 K to temperatures of only 40 K leads to the appearance on new EPR peaks. Warming further leads to a relative increase in the new peaks, and recooling causes them to disappear - the process is completely reversible. At this point the relations of Itoh[6] prove especially useful, for they provide a method of predicting not only the quintet zfs parameters, but also those of the triplet state of the tetraradical. As discussed below, the triplet is the most likely candidate for a thermally excited state of quintet **3**, and indeed we find that the new peaks that appear on warming are where one would expect the outer lines of the triplet spectrum to appear. We thus conclude that the quintet and triplet (and singlet - see below) states of **3** are in thermal equilibrium even at quite low temperatures. We will return to this observation after describing our results for **4**.

Photolysis of the bisdiazene precursor to **4** gives qualitatively different results than those seen with **1-3**. While short photolysis times produce the expected monodiazene TMM, extended photolysis produces no new signals. Instead, the triplet biradical signal starts to weaken in intensity irreversibly. The photochemistry of the diazene precursors to TMMs is absolutely reliable – a very large number of structures have been prepared by this route. There is no reason to even suspect that it could fail for the precursor to **4**. Thus, we conclude that extended photolysis does lead to loss of the second N_2, but that the tetraradical has a *singlet* ground state.

Energy Spectrum

The bis(TMM) design allows some clear-cut predictions concerning the relative energies of the various spin states of the tetraradicals. For a generic tetraradical (four electrons in four nearly degenerate, non-bonding molecular orbitals (NBMO)), one expects 36 states: 20 singlets, 15 triplets, and 1 quintet. However, the strong triplet preference of the TMM unit rules out involvement of states in which these building blocks are not acting at triplets. This greatly simplifies the problem, giving only three states: one singlet, one triplet, and one quintet. Furthermore, if the triplet preference of the TMM is much stronger than the coupling preference (either singlet or triplet) of the potential ferromagnetic coupling unit, then a simple Heisenberg Hamiltonian should be applicable. This leads to only two possible energy patterns, as illustrated in Figure 4.

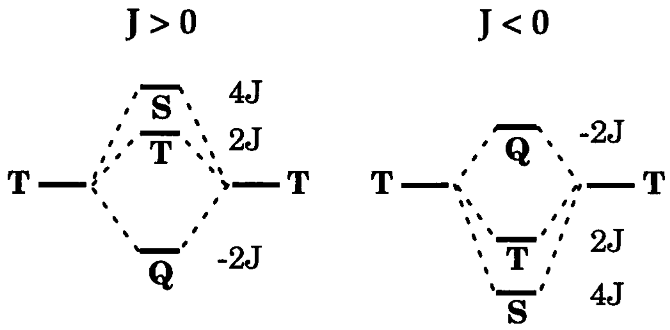


FIGURE 4 The Heisenberg energy levels for tetraradicals such as **1** - **4**.

We can now discuss the results for **1-4** with reference to this Figure. For **1** and **2**, only a quintet is seen, with no thermal population of a nearby triplet. This indicates that J is large and positive. For **3** we see a quintet ground state, but at elevated temperatures the triplet becomes thermally accessible. If we fit the variation in signal intensity with temperature to a Heisenberg model, we get a good fit to J = 50 cal/mol. Finally, for **4** we see no population of any triplet or quintet state of the tetraradical. This leads to an admittedly less firm conclusion, in that it is based on what is essentially negative evidence. Nevertheless, these systems are so well behaved in a variety of contexts, that we feel confident that J is large and negative for **4**.

What would one have expected for these systems? As noted, **1-3** are based on biradicals that are known to have triplet ground states, and so one might have expected ferromagnetic coupling in the tetraradicals. No experimental data for a cyclohexanediyl exist. However, high quality *ab initio* calculations on simple biradicals might be expected to produce meaningful results, and Table 1 summarizes the relevant calculations. Qualitatively, the results are completely in accord with our observations. The biradicals corresponding to **1-3** all have triplet ground states, and that corresponding to **4** has a singlet ground state.

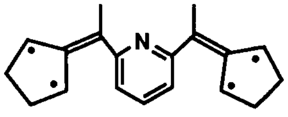
What about quantitative agreement? What would one expect the J to be in tetraradical **3**? For the biradical (**6**), the singlet-triplet gap is 900 cal/mol. In the Heisenberg model, the gap in a biradical is 2J, and so J = 450 cal/mol for the 5-ring system. This should translate directly to **3**, with one modification. In the biradical, 100% of the spin of the two monoradicals is on the cyclopentane carbons. In **3**, only $\frac{1}{3}$ of the spin density of each TMM unit is on the cyclopentane carbon. So, the effective J should be scaled by $\frac{1}{3} \bullet \frac{1}{3}$. This produces for **3**, J = 450/9 = 50 cal/mol, in exact agreement with experiment. Likewise, the larger magnitudes of the J values for the other ferromagnetic coupling units is consistent with our failure to observe thermal population of any other than the ground spin state. Of course, the precision of the agreement in the case of **3** must be fortuitous to some extent, but these results do indicate that the bis(TMM) approach offers a way to evaluate ferromagnetic coupling units qualitatively and quantitatively.

A valuable "spin off" of the bis(TMM) approach is that it provides useful information on the simple biradicals that comprise the ferromagnetic coupling units. We would consider our determination of a singlet ground state for **4** as direct evidence that chair cyclohexanediyl has a singlet ground state. This is consistent with the calculations of Table 1, and also with our model for singlet-triplet gaps in 1,3-biradicals, which emphasizes the role of through-bond coupling.[7] Also, our determination of J = 50 cal/mol for **3** can be considered as a measurement of J for the cyclopentanediyl, after scaling by a factor of 9.

New Systems

The generality of the bis(TMM) approach and the considerable synthetic flexibility associated with it allows one to design tests of many theoretical predictions concerning spin-spin interactions. We are pursuing this line of inquiry along several directions. Here, we will briefly outline our current targets and summarize some preliminary observations.

Substituent effects on spin-spin interactions are a topic of current interest. For example, Yamaguchi has made the theoretical prediction that introduction of charge could reverse normal spin state orderings in a polyradical.[8] This is an intriguing suggestion, especially with regard to the polaronic ferromagnet (see below), wherein the doping process invariably introduces charges. In the one test to date by Itoh, as described in the current proceedings, a reversal was not seen. We can address these issues quite easily with the bis(TMM) approach, and structures **7-9** show our first targets. The pyridine introduces a polar, but uncharged, group, while phenoxide **9** introduces charge. In both cases the polar substitution is at an active (starred) position of the quintet.



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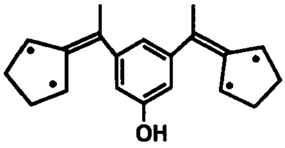

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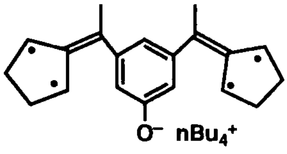

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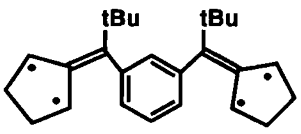
We have prepared the bisdiazene precursors to all three tetraradicals 7-9, and observed a quintet structure in each case. For 7, there is a quite noticeable increase in the zfs D value, consistent with what has been seen in some other heteroatom derivatives of non-Kekulé molecules. Our ultimate goal with 7 is to make the N-oxide and then the N^+-methyl compound as a way to progressively increase polar character. However, we have yet to synthesize the appropriate bisdiazenes.

The phenol 8 and the phenoxide 9 have thus far been studied only at 77 K. In each case, however, a clean quintet spectrum that is qualitatively identical to that for 1 is seen. It will be interesting to see if variable temperature studies reveal any new effects in 9. We also have plans to introduce a positively charged group in place of the phenoxide, in order to further test whether charge can alter spin.

Another topical problem is the effect of twisting out of conjugation, especially with regard to the m-phenylene ferromagnetic coupling unit. This is easy to test with the series 1, 10, 11, and we have prepared all the tetraradicals. Molecular mechanics calculations on the analogous fulvenes have been used to probe the twist angle, Φ, in the tetraradicals. Interestingly, even the "parent" system 1 is quite twisted, with $\Phi \approx 45°$. A t-butyl substituent makes $\Phi \approx 90°$, and so in 10 both TMMs are out of conjugation with the ferromagnetic coupling unit, while in 11, one is in conjugation and one is out.



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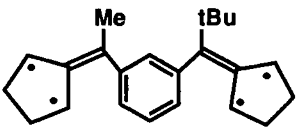

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Photolysis of the bisdiazene precursor to 10 produces only a TMM triplet, which does not diminish in intensity with extended photolysis. We tentatively interpret this to mean that 10 consists of two, non-interacting triplets, in contrast to some other studies which would predict a singlet ground

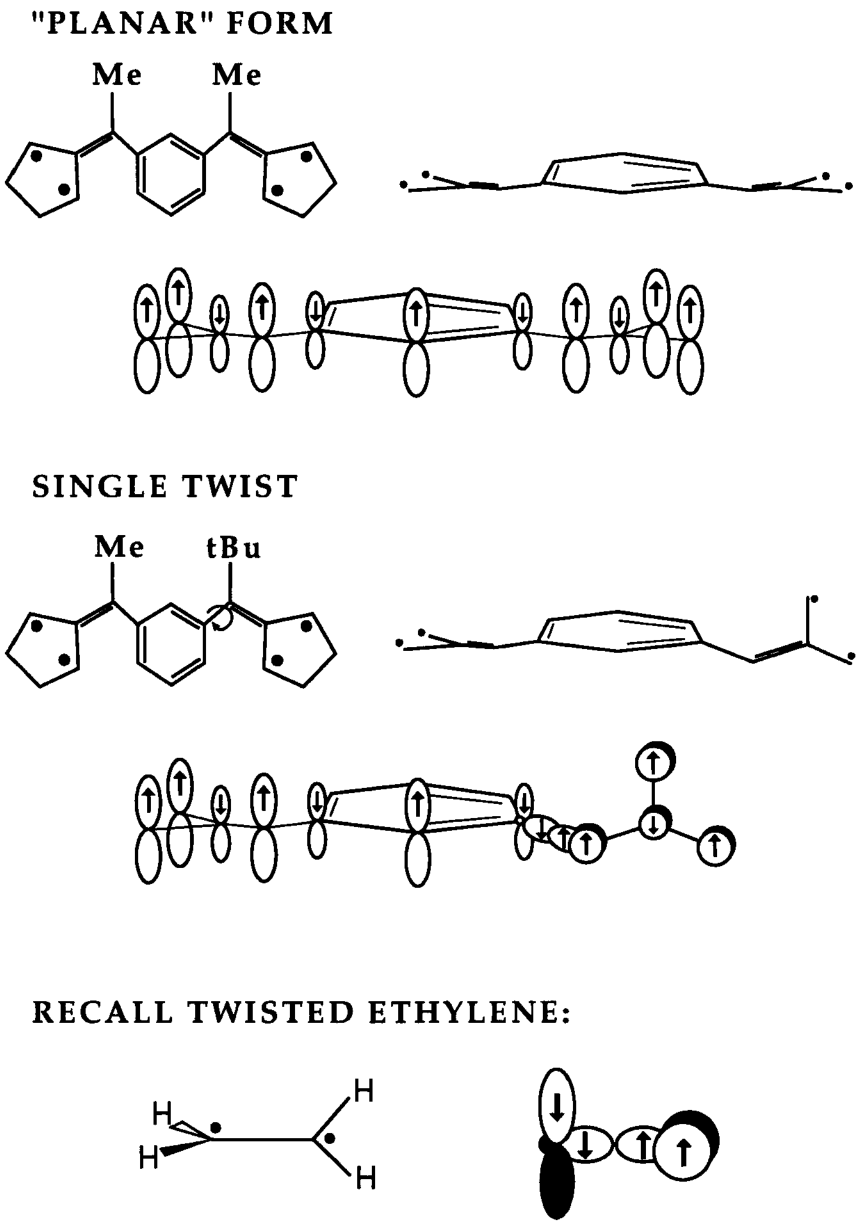
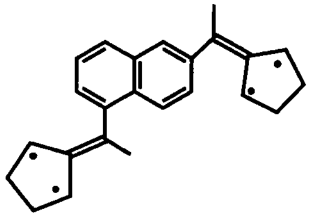


FIGURE 5 Effect of twisting on spin coupling in a m-phenylene unit.

state for **10**. The spectra produced on photolysis of the bisdiazene precursor to **11** are complex, but our current conclusion is that a quintet is produced. Our interpretation of this observation is illustrated in Figure 5. Recall that ethylene, when twisted 90° out of planarity, still has a singlet ground state. This is ascribed to a spin polarization effect, mediated by the still intact single bond. A similar analysis can be applied to **11**. The in-plane TMM will induce a polarization in the m-phenylene π system, as shown in the Figure. Then, polarization of the σ bond that connects to the twisted TMM leads to a polarization of its π system as shown and, ultimately, a quintet ground state. Preliminary data suggest that the quintet state of **11** is in equilibrium with a triplet state, but further work will be required to prove this. If it is correct, however, this could provide an opportunity to develop a realistic estimate of the singlet-triplet gap in twisted ethylene, along the lines of the analysis for **3**.

Finally, we note that the bis(TMM) strategy allows the evaluation of more complex ferromagnetic coupling units, including again structures for which the analogous biradicals would be very difficult to prepare. Preliminary results indicate the naphthalene system **12** has a quintet ground state, as expected based on a */non-* analysis of the appropriate biradical. The azulene structure demonstrates the potential of the bis(TMM) approach to evaluate non-alternant systems, for which simple topology rules have not been developed. We have prepared the ferrocene TMM **14** and found that its zfs value is $|D/hc| = 0.031\ \mathrm{cm}^{-1}$. This is larger than any simple TMM, and may indicate significant contributions from spin-orbit terms from the iron. Encouraged by this indication that the TMM and the iron of **14** do interact, we prepared tetraradical **15** to evaluate ferrocene as a novel ferromagnetic coupling unit. However, preliminary indications are that the TMMs of **15** do not interact appreciably.



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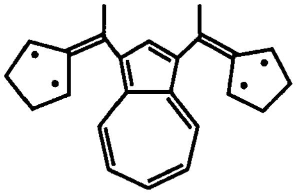

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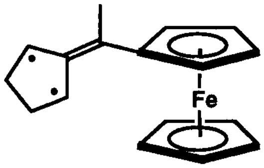

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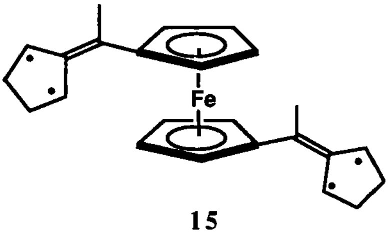

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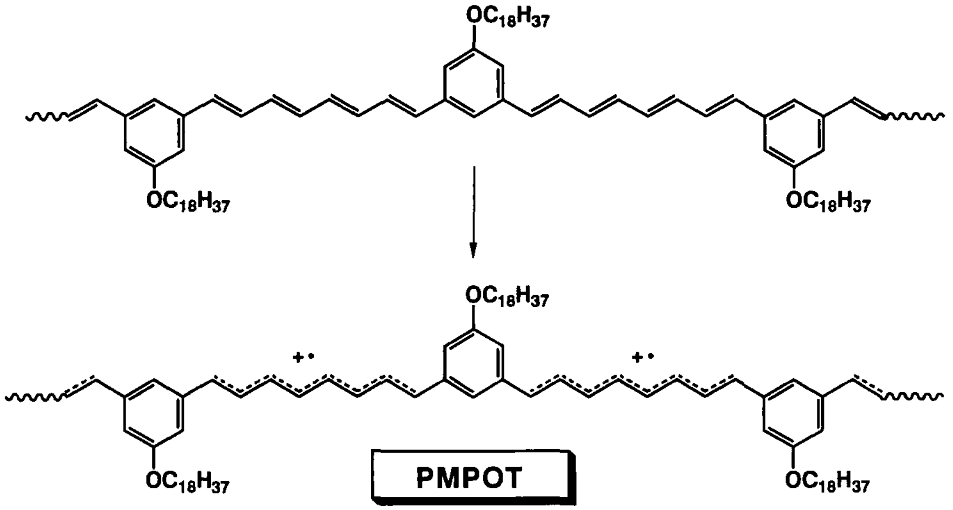
THE POLARONIC FERROMAGNET

The scheme of Figure 1 contains two building blocks, and the bis(TMM) strategy allows and evaluation of potential ferromagnetic coupling units. The other issue is the spin containing unit, and, among other strategies, we have been pursuing the so-called polaronic ferromagnet. Some time ago, theoretical work by Fukutome[9] suggested a design for magnetic materials that, when cast in the model of Figure 1, consists of polarons linked by ferromagnetic coupling units. Recall that a polaron is a radical cation (anion) obtained by oxidative (reductive) doping of a conjugated polymer. From our perspective, there are several advantages to this approach. From the conducting polymer literature we know that polarons can be stable at room temperature, and that they can be readily introduced by a variety of methods. This latter feature circumvents the often daunting challenge of synthesizing polymers with a large number of spins.

The polaronic ferromagnet is one of several polymer strategies we are pursuing, and have presented our major results to date on this problem elsewhere.[10] Here we will summarize the current status of the problem and speculate on future challenges.

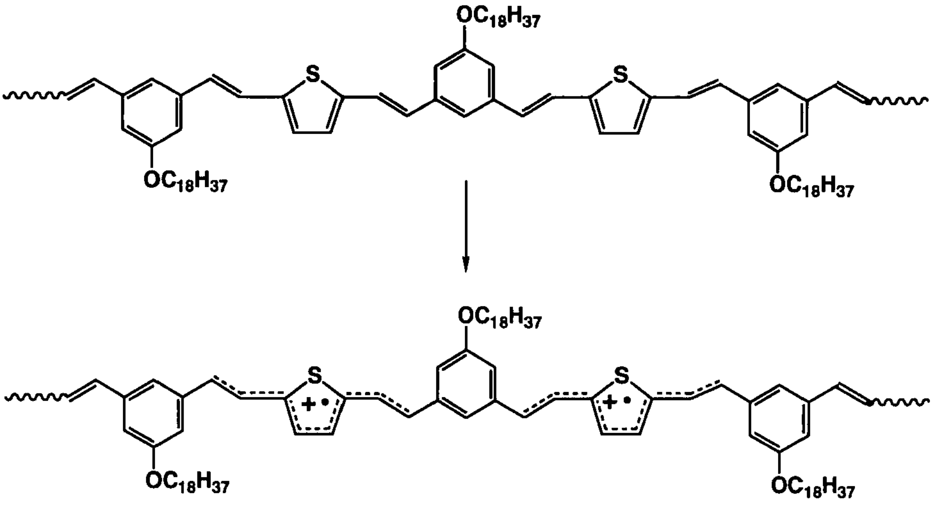
Our initial target in this system was the polymer poly(m-phenyleneoctatetraene) (PMPOT), shown below. The polymer could be synthesized with surprising efficiency by a Wittig reaction, with the octadecyl side chain playing the crucial role of keeping the polymer in solution so that long chain lengths can develop. On doping, a significant number of spins are introduced, and these spins are *stable at room temperature.*

Magnetic measurements on PMPOT revealed that it was a paramagnet. However, saturation behavior was not that of a spin $\frac{1}{2}$ system, which would be expected for a system consisting of isolated polarons. Instead, the saturation behavior fit to a Brillouin function with S between 2.0 and 2.5. Note that one should not place too much emphasis on the exact value of S – it is no doubt an average of a distribution a spin states that might be quite skewed. That is, S = 2.5 might indicate a Gaussian or similarly smooth distribution of spin states, centered around 2.5. However, it could equally well signify that 95% of the sites are $S = \frac{1}{2}$ and 5% are $S = 19$. The two possibilities would display qualitatively similar saturation behaviors. The important point is simply that our measured S is unambiguously greater than $\frac{1}{2}$. We take this to indicate significant ferromagnetic coupling among the polarons, presumably mediated by the m-phenylene ferromagnetic coupling unit.



We were very much encouraged by this initial result, and set out to improve upon the system. Of course, a necessary modification would be the introduction of inter-chain ferromagnetic coupling, since variable temperature studies of PMPOT suggested a weak antiferromagnetic coupling between the chains (as expected). However, before addressing this problem, we set out to improve one significant deficiency of the PMPOT system. Although a precise number is somewhat difficult to obtain, it is clear that the number of spins in our doped polymers is quite low – certainly less than 1% of the monomer units have a spin. Our initial interpretation of this result was that the polarons we were generating were unstable, and many reacted with the surrounding polymer. Consistent with this was the finding that when we coated films of PMPOT on glassy carbon electrodes and attempted electrochemical doping, all oxidation waves were irreversible.

Since electrochemical doping was one of our major goals for this project, we sought to modify the polyene segment, so that more stable polarons could be formed. Borrowing from the conducting polymer literature, we introduced a thiophene unit to produce PMPTV. Our initial results with this polymer were quite encouraging. Stable spins are produced and the saturation behavior is now even more dramatic – S values as high as 4.5 are seen. However, our best estimates still suggested that less than 1% of the monomers have spin.



PMPTV

These results caused us to question one of our fundamental assumptions. We believed that the dopable segments of these polymers would be too short to support bipolaron formation (oxidation to a spinless dication). Our early doping studies did suggest that bipolarons were not forming, in that the spin concentration from doping rises and then levels off to a constant value. If bipolaron formation we important, one would see a rise in spin concentration (polaron formation) followed by a decline (bipolaron formation). We took this to mean that bipolaron formation was not a problem is these systems. However, we did always notice that the mass uptake of dopant was much more than expected based on the number of spins. We took this to indicate clustering of the dopant (i.e., instead of AsF_6^-counterions, one has, for example, $As_2F_{11}^-$ or $As_3F_{16}^-$). However, we now suspect that bipolaron formation may be important.

In order to evaluate the potential importance of bipolaron formation we have launched a theoretical effort to evaluate relevant structures, as summarized in Figure 6. Basically, the method involves PPP calculations with very extensive CI in the π system. "Control experiments" reveal that this method obtains quite reasonable singlet triplet gaps for a number of structures that have been treated at higher levels (TMM, m-xylylene, non-Kekulé benzene, cyclobutadiene, benzene dication).

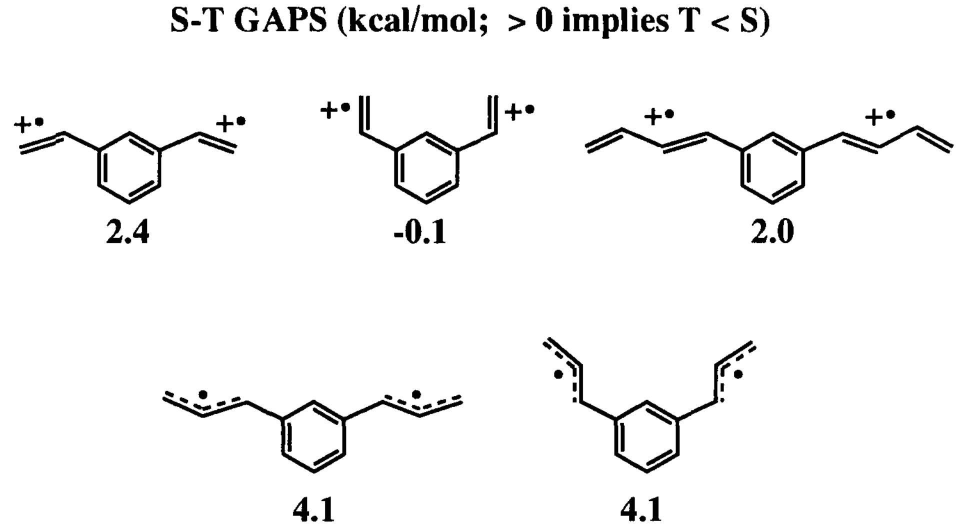


FIGURE 6 PPP-CI singlet triplet gaps.

The calculations of most direct relevance to the polaronic question are those on the various radical cations. The simplest system, just divinylbenzene dication, shows a triplet ground state in the "extended" conformation, but essentially a singlet-triplet degeneracy for the "cisoid" form. This is a surprising result, and it suggests that conformational issues may be more important than had been anticipated previously. Our preliminary analysis of this result suggests that it may be due to an adverse coulombic repulsion between the ethylene unit and C4 of the benzene in the singlet state of the *extended* conformation. This destabilizes the singlet and produces a triplet ground state.

An encouraging result is that the butadiene analogue, in the extended conformation, shows a singlet-triplet gap that is almost equal to the divinyl system. This indicates that the ferromagnetic coupling does not fall off sharply as one increases delocaliztion and, presumably, decreases spin density on the ferromagnetic coupling unit.

An interesting comparison is to the simplest neutral analogue, the diallyl system. This structure shows a triplet ground state regardless of conformation, consistent with the coulombic arguments presented above. It is also clear that, at this level of theory, the diallyl system shows a larger triplet preference than the cation. To first order, each structure has 1/2 spin on the carbon adjacent to the benzene ring, and so, by analogy to the arguments presented above, one might have expected equal singlet-triplet gaps. The calculations suggest that there may be something intrinsically favorable in having a neutral radical (odd number of carbons) rather than a radical ion (even number of carbons) as the spin-containing unit.

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