previously obtained as bacterial metabolites of the parent arenes by *P. putida*, but in many cases, $[\alpha]_D$ values, ee values, and absolute configurations (**3A**, **5A**, **6A**) were unreported.

The chiral lanthanide shift reagent Eu(hfc)$_3$ has proved to be very effective in the determination of ee values of a range of diols,[22] including the dihydrodiols of polycyclic arenes and azarenes (e.g., naphthalene and quinoline). By this method distinguishable ^{1}H NMR signals were found for each enantiomer.[23] When a chemically synthesized racemic sample of the monocyclic *cis*-dihydrodiol **1A** and an enzymatically formed sample of the *cis*-dihydrodiol **3A** of low optical purity (table) were similarly analyzed,[23] the ^{1}H NMR signals for enantiomers were found to be indistinguishable at all concentrations of Eu(hfc)$_3$. The present report illustrates how this problem can be circumvented in the *cis*-dihydrodiol series by synthesis of appropriate diesters from α-methoxy-α-(trifluoromethyl)phenylacetic acid (MTPA).

Initial attempts to form di-MTPA esters of the *cis*-dihydrodiols **1A–6A** resulted in their total aromatization. *cis*-Diols **1A–6A** were, however, found to react with 4-phenyl-1,2,4-triazoline-3,5-dione at ambient temperature (12 h) to give cycloadducts **1B–6B**. ^{1}H NMR analysis of the crude samples of **1B–6B** showed essentially a single diastereoisomer (>97%) to be present. Pure diastereoisomers **1B–6B** were obtained by PLC purification in ca. 70–80% yield. Treatment of the cycloadducts **1B–6B** with the acid chloride of (+)-(*R*)-MTPA in pyridine yielded the corresponding di-MTPA esters (**1C**$_R$–**6C**$_R$) in quantitative yield (table).

The *cis*-dihydrodiol metabolite **1A** ($[\alpha]_D$ +25.6°, MeOH) had previously been shown to be optically pure and of the 1*S*,2*R* configuration and thus the di-MTPA ester **1C**$_R$ has the R′,-*R″*,1*S*,2*R* configuration (*R′* and *R″* refer to the absolute configurations of the MTPA groups in the di-MTPA ester.). Since the 1*R*,2*S* enantiomer of *cis*-dihydrodiol **1A** was unavailable in sufficient quantities either as a metabolite or by synthesis,[23] it was impossible to synthesize the corresponding (*R′*,*R″*,1*R*,2*S*)-di-MTPA diastereoisomer in order to observe the characteristic δ values in the ^{1}H NMR spectrum that might distinguish it from the **1C**$_R$ configuration. However, diesterification of the cycloadduct **1B** derived from the 1*S*,2*R* enantiomer of *cis*-dihydrodiol **1A** with the acid chloride of (−)-(*S*)-MTPA yielded the di-MTPA ester of *S′*,*S″*,1*S*,2*R* configuration (**1C**$_S$), which is the enantiomer of the elusive *R′*,*R″*,1*R*,2*S* diastereoisomer and hence spectrally indistinguishable from it. The diagnostic δ values for diastereoisomers **1C**$_R$ and **1C**$_S$ are thus identical with those that could be found for the di-MTPA esters derived from each enantiomer of **1A** by using a single enantiomer of MTPA. Use of both the respective acid chlorides of (+)-(*R*)- and(−)-(*S*)-MTPA in the diesterification of the cycloadducts of *cis*-dihydrodiols **2A**, **4A**, **5A**, and **6A**, again showed each sample to be homochiral. The distinguishable MeO ($\delta_{^1\mathrm{H}}$) and CF$_3$ ($\delta_{^{19}\mathrm{F}}$) signals in the NMR spectrum of each di-MTPA diastereoisomer obtained from the cycloadduct **3B** of *cis*-diol **3A** with the acid chloride from either (+)-(*R*)-or (−)-(*S*)-MTPA allowed a value of ca. 60% ee to be estimated (table). Fractional recrystallization of **3B** yielded the major enantiomer in pure form. It has also proved possible to use this method in the determination of optical purity of the *cis*-dihydrodiols of naphthalene and quinoline after catalytic hydrogenation to yield *cis*-tetrahydrodiols followed by diesterification using both (+)-*R* and (−)-*S* forms of MTPA.[23]

X-ray crystal structure analysis was carried out on suitable crystals of compounds **1C**$_S$, **3C**$_R$, and **5C**$_S$, and it revealed that the cycloaddition reaction between the *cis*-dihydrodiols **1A**, **3A**, and **5A** and the triazolinedione dienophile had occurred almost exclusively (>97%) syn to the hydroxyl groups (yielding essentially the single diastereoisomers **1B**, **3B**, and **5B**). Previous cycloadditions on the diester or acetonide derivatives of *cis*-dihydrodiols (of structure similar to compounds **1A**, **3A**, and **5A**) using a range of dienophiles often yielded two or four diastereoisomers from each enantiomer, i.e., exo and endo isomers of both anti[21,24–27] and syn[21,27] cycloadducts.

Since the absolute configuration of the MTPA group for the diesters **1C**, **3C**, and **5C** is known, the configurations of the other chiral centers and of the parent *cis*-dihydrodiols **1A**, **3A**, and **5A** can be unequivocally established (table). The 1*S*,2*R* configuration is established for metabolite **5A** and is also confirmed for metabolite **1A**. The major enantiomer found in the metabolite **3A** is now assigned a 1*S*,2*S* configuration.

The absolute configuration of each *cis*-dihydrodiol enantiomer [now firmly established by X-ray crystallographic (**1A**, **3A**, **5A**) and other[1–5] methods (**1A**, **2A**, **4A**)] was associated with several diagnostic ^{1}H and ^{19}F NMR signals (500 MHz, CDCl$_3$) in the respective di-MTPA esters (**1C–6C**). Thus, a *cis*-dihydrodiol having a 1*S* configuration gave a di-MTPA ester that was characterized by (i) the downfield MeO signal having a larger positive $\delta_{^1\mathrm{H}}$ value (3.61–3.65 ppm) and (ii) the downfield CF$_3$ signal having a smaller negative $\delta_{^{19}\mathrm{F}}$ value (−7.66 to −8.39 ppm) when the (+)-*R* enantiomer of MTPA was used. Conversely, the di-MTPA ester from a *cis*-dihydrodiol having a 1*R* configuration shows a smaller δ_1 value (3.21–3.55 ppm) and a larger negative δ_{19} value (−8.72 to −9.28 ppm) for the corresponding signals when (+)-(*R*)-MTPA is utilized.

The applicability of this method to *cis*-dihydrodiols of monocyclic arenes bearing several substituents is currently under investigation.

Acknowledgment. We thank the Department of Education for Northern Ireland and ICI plc for a CAST award (to M.V.H.), the Biotechnology Directorate of SERC (to N.D.S. and D.J.G.) and the Queen's University of Belfast (to M.R.J.D.) for financial support, and Dr. Paul Stevenson for helpful advice and assistance.

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Synthesis of the First 2,2′-Biphosphinine. X-ray Crystal Structure Analysis of Its Tetracarbonylchromium Complex

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2,2′-Bipyridines have found widespread use in analytical and synthetic coordination chemistry over many decades.[1] Complexes such as [Ru(bipy)$_3$]$^{2+}$ have special redox and photocatalytic properties[2] which have been the subject of extensive studies. In view of that situation, the design of phosphorus analogues of bipyridines was an attractive goal. Some time ago, we synthesized a monophosphorus analogue **1**[3] and performed a preliminary investigation of its coordinating properties.[4]

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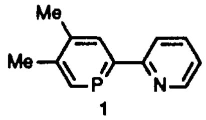
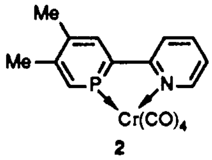
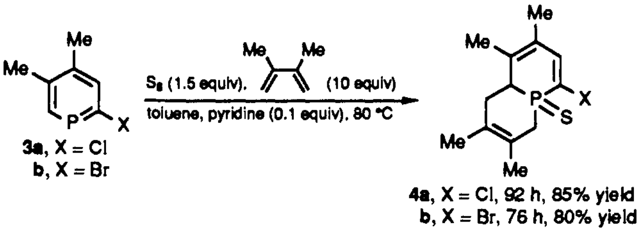


1



2

The chelate **2** was characterized by X-ray crystal structure analysis.[4] As a logical next step, we decided to synthesize the symmetrical 2,2′-biphosphinines. We used the easily available 2-halophosphinines[5] as starting material. Direct coupling of these 2-halophosphinines using Ni(0) species [$NiCl_2$, Zn, PPh_3, DMF] was completely unsuccessful: nickel complexes of 2-halophosphinines were formed. The stabilizing interaction between the π-aromatic system and the halogen substituent appeared to be so strong that it was found necessary to destroy this aromaticity in order to reactivate the C–X bonds. The combined reaction of sulfur and 2,3-dimethylbutadiene as in a preceding work[6] proved to be the solution (eq 1).

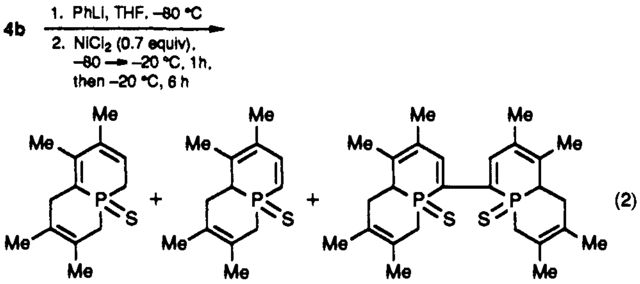


3a, X = Cl
b, X = Br

4a, X = Cl, 92 h, 85% yield
b, X = Br, 76 h, 80% yield

(1)

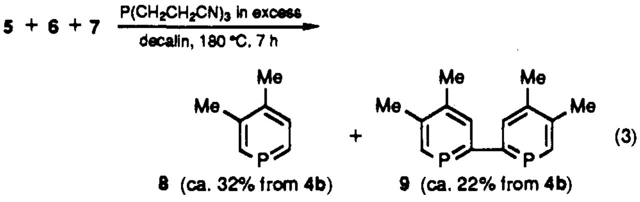
Pyridine was used as a catalyst, and the [4 + 2] cycloaddition selectively took place on the unsubstituted P=C bond.[7] The bromo compound **4b** proved to be sufficiently reactive to undergo a bromine–lithium exchange by reaction with phenyllithium. The resulting 2-lithio derivative was then treated with $NiCl_2$. A mixture of protonation and coupling products[8] was thus obtained (eq 2).



5 (ca. 35%) **6** (ca. 10%) **7** (ca. 35%)

(2)

The chromatograhic separation of **5**, **6**, and **7** is difficult. Thus, the mixture was directly reduced by using a trialkylphosphine[9] (eq 3). At room temperature, $P(CH_2CH_2CN)_3$ and its sulfide crystallize out and **8** and **9** are easily separated by chromatography on silica gel with hexane. The monophosphinine **8** has already been described elsewhere.[10] The biphosphinine **9**[11] was completely characterized by ^1H, ^{13}C, and ^{31}P NMR and mass spectrometry. The simulation of the ^{13}C NMR spectrum was carried out. The best fit was obtained with $^3J(P-P) = 57.6$ Hz. The only special feature of this spectrum concerns C_3 and C_3', which appear as singlets. The $Cr(CO)_4$ chelate **10**[12] was then prepared as described in eq 4. The X-ray crystal structure analysis of **10** (Figure 1) revealed that the molecule is almost perfectly symmetrical. The two phosphinine rings are strictly coplanar, and two carbonyls



8 (ca. 32% from **4b**) **9** (ca. 22% from **4b**)

(3)

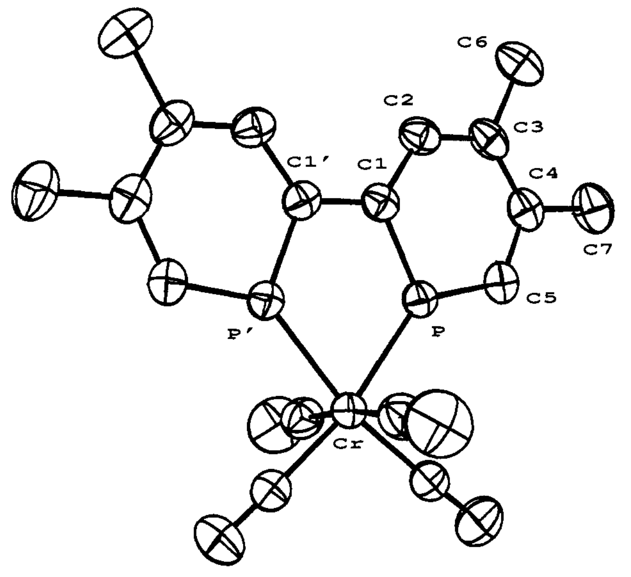
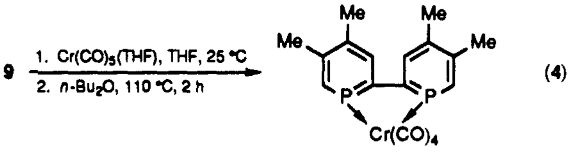


Figure 1. ORTEP drawing of one molecule of **10**. Vibrational ellipsoids are scaled to enclose 50% of the electron density. Hydrogen atoms are omitted for clarity. Principal bond distances (Å): Cr–P, 2.2952 (7); $Cr-C_8$, 1.894 (3); $Cr-C_9$, 1.865 (3); $P-C_1$, 1.737 (2); $P-C_5$, 1.711 (3); O_8-C_8, 1.138 (4); O_9-C_9, 1.144 (3); C_1-C_1', 1.467 (3); C_1-C_2, 1.392 (3); C_2-C_3, 1.395 (4); C_3-C_4, 1.400 (3); C_4-C_5, 1.385 (4). Selected bond angles (deg): P–Cr–P′, 77.15 (3); $Cr-P-C_1$, 117.74 (8); $Cr-P-C_5$, 137.93 (9); C_1-P-C_5, 104.3 (1); $P-C_1-C_1'$, 113.7 (2); $P-C_1-C_2$, 120.4 (2); $C_1'-C_1-C_2$, 125.9 (2); $C_1-C_2-C_3$, 126.1 (2); $C_2-C_3-C_4$, 122.1 (2); $C_3-C_4-C_5$, 123.2 (2); $P-C_5-C_4$, 123.9 (2).



10 (80%)

(4)

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(7) Sulfides **4a** and **4b** were purified by column chromatography with hexane as eluent to remove excess sulfur and finally with hexane/ether (3:1). ^{31}P NMR ($CDCl_3$, 32.44 MHZ): **4a** δ 31.26 ppm; **4b** δ 30.30 ppm.

(8) Compounds **5**, **6**, and **7** were purified by chromatography with hexane/ether (3:1) as eluent. ^{31}P NMR (CH_2Cl_2): **5** δ 11.61 ppm; **6** δ 22.07 ppm; **7** δ 22.30 ppm.

(9) $P(CH_2CH_2CN)_3$ is available in large quantities from Hoechst. It has been chosen for its stability in air and its poor solubility in organic solvents at room temperature.

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(11) The reaction mixture was chromatographed with hexane to remove decalin and 4,5-dimethylphosphinine and finally with hexane/benzene (5:1) to give **9**: ^{31}P NMR ($CDCl_3$) 178.32 ppm; ^1H NMR ($CDCl_3$) δ 2.36 (s, 12 H, 4 Me), 7.83 (d, 2 H, AA′XX′, $|^3J(H-P_A) + {}^4J(H-P_B)| = 2.6$ Hz, $H_{3,3'}$), 8.37 (q, 2 H, AA′XX′, $|^2J(H-P_A) + {}^5J(H-P_B)| = 39.19$ Hz, $H_{6,6'}$) ppm; ^{13}C NMR ($CDCl_3$) δ 22.65 (s, Me), 23.06 (s, Me), 136.27 (s, $C_{3,3'}$), 139.66 (AXX′, $|\sum J(C-P)| = 15.75$ Hz, $C_{4,4'}$ or $C_{5,5'}$), 142.30 (AXX′, $|\sum J(C-P)| =$ 13.42 Hz, $C_{4,4'}$ or $C_{5,5'}$), 154.93 (AXX′, $|^1J(C-P_A) + |^4J(C-P_B)| = 48.83$ Hz, $C_{6,6'}$), 170.40 (AXX′, $|^1J(C-P_A) + {}^2J(C-P_B)| = 24.2$ Hz, $C_{2,2'}$) ppm; C_6 and C_2 were simulated independently, with J(PC) estimated initially from their values in **1**.[4] Subsequent iteration (giving $^1J(P-C) = -52.4$ Hz, $^4J(P-C) =$ 2.8 Hz for C_6, and $^1J(P-C) = -50.5$ Hz, $^2J(P-C) = 26.3$ Hz for C_2) converged upon $^3J(P-P)$ of 58.1 and 57.6 Hz with ^{13}C isotope shifts upon adjacent ^{31}P nuclei of 8.5×10^{-2} and 7.9×10^{-2} ppm, respectively. Mass spectrum m/e (rel intensity) 246 (M, 100), 231 (M − 15, 38).

(12) Complex **10** was purified by column chromatography with hexane/CH_2Cl_2 (3:1) as eluent. **10**: ^{31}P NMR (THF) δ 227.17 ppm; ^{13}C NMR ($CDCl_3$) δ 22.79 (s, Me), 24.11 (d, $J(C-P) = 4.42$ Hz, Me), 130.13 (AXX′, $|^2J(C-P_A) + {}^3J(C-P_B)| = 21.16$ Hz, $C_{3,3'}$) 136.62 (d, $J(C-P) = 24.12$ Hz, $C_{4,4'}$ or $C_{5,5'}$), 146.65 (AXX′, $|^2J(C-P_A) + {}^5J(C-P_B)| = 24.85$ Hz, $C_{4,4'}$ or $C_{5,5'}$), 147.83 (AXX′, $|^1J(C-P_A) + {}^4J(C-P_B)| = 20.06$ Hz, $C_{6,6'}$), 158.92 (AXX′, $|^1J(C-P_A) + {}^2J(C-P_B)| = 60.10$ Hz, $C_{2,2'}$), 213.22 (AXX′, $|^2J(C-P_A) + {}^2J(C-P_B)| = 34.49$ Hz, CO equatorial), 227.65 (AX_2, $^2J(C-P) = 7.85$ Hz, CO axial) ppm; mass spectrum m/e (rel intensity) 410 (M, 13), 354 (M − 2CO, 4), 326 (M − 3CO, 15), 298 (M − 4CO, 100), 246 (M − $Cr(CO)_4$, 22).

(C_9O_9, $C_9'O_9'$) practically lie in this plane: $C_9CrC_9'/PC_1C_1'P'$ interplane angle 6.46° ± 0.4°. Conversely, the plane of the two other carbonyls (C_8O_8, $C_8'O_8'$) is almost orthogonal to the biphosphinine plane (94.94° ± 0.05°). When the structures of **2** and **10** are compared, several significant differences appear. Whereas the two $P\overset{..}{-}C$ and two $N\overset{..}{-}C$ bonds of **2** are almost strictly equal (1.707 and 1.709 Å; 1.367 and 1.358 Å) as in (bipy)Cr$(CO)_4$[13] indicating that the two aromatic rings are not disturbed by the bridge, the two internal $P\overset{..}{-}C$ bonds of **10** are notably longer than the two external ones (1.737 vs 1.711 Å). Theoretical calculations are currently underway to establish whether or not delocalization occurs in the five-membered metallacycle of **10**. In addition, we are trying to improve the yield of the bridging step (eq 2), and we are starting to investigate the coordination chemistry of **10**.

Supplementary Material Available: ^1H NMR, ^{13}C NMR, and mass spectra of **4a,b** and **5-7**, X-ray structural determination for **10** including experimental details, and tables of bond distances and angles and their estimated standard deviations, positional parameters and their estimated standard deviations for all nonhydrogen atoms, and β_{ij} with their estimated standard deviations for **10** (6 pages). Ordering information is given on any current masthead page.

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(13) Main data for (bipy)Cr$(CO)_4$: N-C, 1.347 (3) Å (mean); C-C bridge, 1.471 (5) Å; N-Cr, 2.089 (3) Å (mean). This structure will be fully described elsewhere.

Cubane Radical Cation in Liquid Hydrocarbons: Time-Resolved Fluorescence Detected Magnetic Resonance Study[†]

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The structure and rearrangement of strained hydrocarbons and radical cations in general, and those of the $(CH)_8$ group in particular, have attracted considerable experimental and theoretical attention.[1-8] The electronic structure of the cubane radical cation (cubane$^{•+}$) is particularly interesting, since the cubane molecule has the highest strain energy of the set and belongs to the O_h point group.[8] The photoelectron spectroscopic study and ab initio STO-3G and MINDO/3 calculations by Heilbronner et al. indicate that cubane has triply degenerate highest occupied molecular orbitals (t_{2g} orbitals).[1] Upon ionization it undergoes Jahn-Teller distortion:[9] the symmetry is lowered from O_h to C_{2v}. An EPR study of cubane photolyzed in a neon matrix at 4 K was reported by Knight et al. in 1987.[2] However, Knight repeated these experiments recently and concluded that cubane$^{•+}$ was not observed in his experiments.[10] In view of this finding, we conclude that the cubane radical cation was not observed previously and that in our studies of cubane$^{•+}$ rearrangements in Freon matrices we could not directly observe the EPR spectrum of cubane$^{•+}$ in several Freon matrices at 77 K. Cubane$^{•+}$ undergoes rearrangement at 80 K and higher temperatures, and one cannot utilize a Freon matrix to stabilize and observe its EPR spectrum in the temperature range (110-120 K) where resolved EPR spectra of radical cations in $CF_2ClCFCl_2$ are usually obtained.[7]

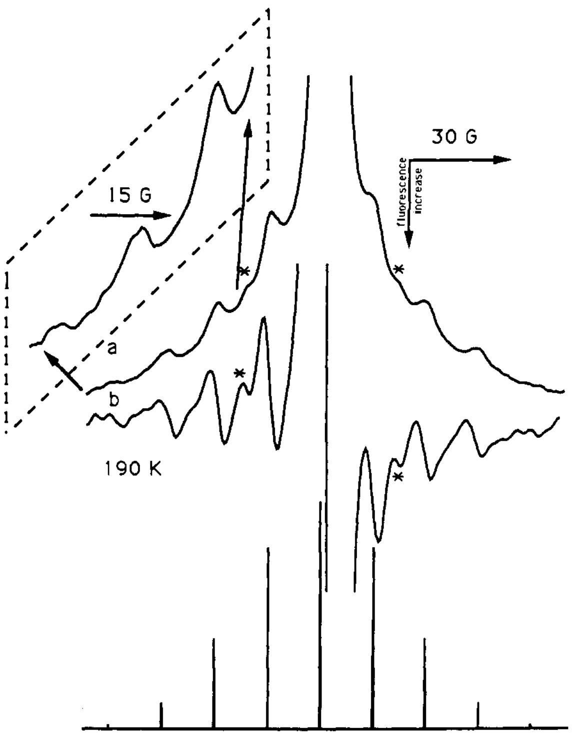

Figure 1. (a) FDMR spectrum observed at 190 K in cyclopentane containing 10^{-3} M cubane and 10^{-4} M perdeuterated anthracene. The asterisks indicate the signals from the cyclopentane solvent.[20] The insertion is the enlargement of the left outermost three peaks. (b) First-derivative FDMR spectrum of part a.

We report here a time-domain study of cubane$^{•+}$, utilizing the time-resolved fluorescence detected magnetic resonance (FDMR) technique.[11] We have observed a radical cation, which we assign to cubane$^{•+}$ in liquid hydrocarbon solution. The observed FDMR spectrum is consistent with the presence of eight protons (a_{8H} = 16.1 G) of the cation which are equivalent.

Cubane$^{•+}$ was produced in cyclopentane solutions 10^{-3} M in cubane and 10^{-4} M in perdeuterated anthracene (A) using pulsed electron beam (12-ns width) irradiation, generated by a 3 MeV electron Van de Graaff generator. The ionizing radiation creates singlet-phased radical ion pairs in cyclopentane (c-C_5H_{10}, gas-phase ionization potential (IP) = 10.4 eV[12]) consisting of a cyclopentane radical cation (c-$C_5H_{10}^{•+}$) and an electron, most of which recombine geminately in a few picoseconds. A small part of c-$C_5H_{10}^{•+}$ reacts by transferring positive charge to dissolved cubane (IP = 9.1 eV[1]) to form cubane$^{•+}$, while some of the electrons are converted to the scintillator radical anion ($A^{•-}$). The resulting radical ion pairs consisting of cubane$^{•+}$ and $A^{•-}$ are still spin-correlated. Upon their recombination, an excited singlet state

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