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Selective Side-Chain Oxidation of Peralkylated Pyrromethene–BF_2 Complexes

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ABSTRACT

Treatment with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) oxidized 2,6-diethyl-1,3,5,7,8-pentamethylpyrromethene–BF_2 complex **1**, *13,14-trimethyl-2, 3, 4, 5,9,10,11,12-octahydroindomethene–BF_2 complex* **5**, *and 1,3,5,7,8-pentamethyl-1,2,3, 5,6,7-hexahydropyrromethene–BF_2 complex* **8** *to the weakly fluorescent 3-formyl, 5-oxo, and 8-formyl derivatives* **4**, **6**, *and* **9**, *respectively. The dye* **1** *was oxidized by lead tetraacetate to 1,7,8-trimethyl-2,6-diethyl-3,5-diacetoxymethylpyrromethene–BF_2 complex* **12** [λ_f *(ethanol) 538 nm,* Φ *0.62,* λ_{las} *(ethanol) 555–570 nm*]. *Catalytic reduction (Pd/C) converted the aldehyde* **4** *to 2,6-diethyl-3-hydroxymethyl-1,5,7,8-tetramethylpyrromethene–BF_2 complex* **10** [λ_f *(ethanol) 537 nm,* Φ *0.70,* λ_{las} *(ethanol) 547–575 nm*].

INTRODUCTION

In 1993, 2,6-diethyl-1,3,5,7,8-pentamethylpyrromethene–BF_2 complex **1**, λ_{las} (ethanol) 567 nm, became the undisputed champion in laser dye activity insofar as it afforded twice the power efficiency obtained from Rhodamine–6G (R–6G), unsurpassed in laser dye activity for over 25 years [1–4]. Other pyrromethene–BF_2 complexes (P–BF_2) also surpassed R–6G in laser activity [1]. Elsewhere, P–BF_2 derivatives have been useful as fluorescent probes in medical and biological research [5] and as photodynamic therapeutic agents for cancer [6]. Apart from their fluorescence, properties of P–BF_2 complexes have received minimal attention. This investigation was undertaken to determine the effect on fluorescence brought about by functionalization of P–BF_2 alkyl substituents. An exploration of the oxidation of alkyl P–BF_2 derivatives treated with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ), and in one instance with lead tetraacetate, was carried out.

Oxidation of an arylalkane by DDQ generally proceeded by an initial abstraction of hydride anion from a benzylic or similar position, leading to a carbenium ion intermediate. An alcohol was produced when the carbenium ion was trapped by water and was, of course, subject to further oxidation to a carbonyl compound. The availability of multiple alkyl substituents generally led to product mixtures with partial regioselectivity determined by initial formation of the most stable carbenium ion [7,8]. A general conversion of methylaryl compounds to aryl acetates by treatment with lead tetraacetate has been known for over 70 years [9].

RESULTS AND DISCUSSION

Alkyl derivatives of P–BF_2 were readily attacked by DDQ. The reaction with 1,3,5,7,8-pentamethyl- and 1,2,3,5,6,7-hexamethylpyrromethene–BF_2 complexes **2** and **3**, examples of P–BF_2 derivatives with unsubstituted positions, led to intractable mixtures. On the other hand, certain peralkylated P–BF_2 compounds were efficiently oxidized to single products.

Treatment with DDQ brought about an oxidation of strongly fluorescent 2,6-diethyl-1,3,5,7,8-pentamethylpyrromethene–BF_2 complex **1** to the weakly fluorescent 3-formyl derivative **4** (80%) and

Dedicated to Prof. Adrian Gibbs Brook on the occasion of his seventieth birthday.

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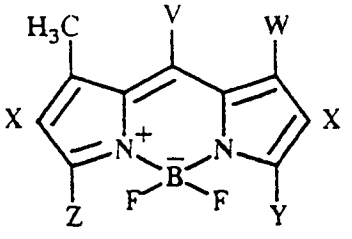
a trace amount of an unidentified mixture. The structure of the aldehyde **4** was confirmed by X-ray crystallographic analysis (Figure 1) [10]. An explanation for this selective oxidation, presumably initiated by hydride abstraction from the 3-alkyl substituent, cannot be offered at this time. In principle, the location of the most stable carbenium ion intermediate at the 3-substituent is subject to theoretical verification by a calculation of delocalization energies [11].

It was assumed that similar oxidations of other peralkylated P–BF_2 compounds by DDQ proceeded by hydride abstraction from a suitable "3-alkyl" substituent and that subsequent trapping with water followed by oxidation gave a "3-acyl" derivative. The single product obtained from a reaction between DDQ and 1,13,14-trimethyl-2,3,4,5,9,10,11,12-octahydroindomethene–BF_2 complex **5** was accordingly assigned the structure of the 5-oxo derivative **6** (47%). Dehydrogenation to 1,13,14-trimethylindomethene–BF_2 complex **7** was not detected [12]. A search for the detection of oxidation of additional alkyl groups in these reactions with P–BF_2 complexes **1** and **5** was unsuccessful.

Hydrogenation (Pd/C) of fluorescent 1,3,5,7,8-pentamethylpyrromethene–BF_2 complex **2** gave the nonfluorescent 1,2,3,5,6,7-hexahydro derivative **8**, an example of a vinamidine–BF_2 complex [13]. In contrast to the destruction of complex **2** on treatment with DDQ, a similar treatment of complex **8** brought about an oxidation to 8-formyl-1,3,5,7-tetramethyl-1,2,3,5,6,7-hexahydropyrromethene–BF_2 complex **9** (65%) and an intractable mixture. A competitive dehydrogenation to restore the complex dye **2**, if it occurred, was presumably followed by its known destruction in the presence of DDQ. An attempt to bring about dehydrogenation of the hexahydro derivative **8** by treatment with DDQ under anhydrous conditions in *o*-dichlorobenzene at 170°C for 16 hours gave an unidentified mixture containing a trace amount of an unidentified fluorescent substance.

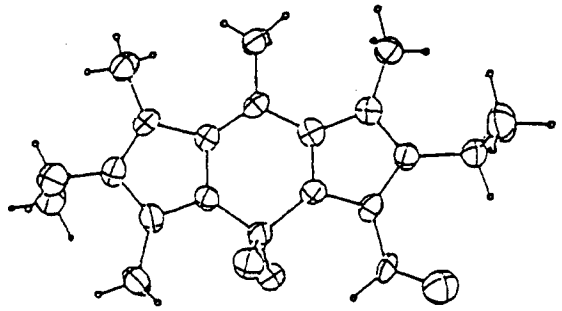
The formation of acyl derivatives **4** and **6** of P–BF_2 complexes offered an illustration of diminished fluorescence in aldehydes and ketones [14]. Nearly 90% of the fluorescence intensity in the laser dye **1**, $\Phi > 0.99$ [3,4], disappeared in its oxidation to complex **4**, Φ 0.12. Fluorescence was largely restored on reduction (Pd/C) of the aldehyde **4** to 2,6-diethyl-3-hydroxymethyl-1, 5, 7, 8-tetramethylpyrromethene–BF_2 complex **10**, Φ 0.70. Other reduction products included the dye **1** and a minor product assigned the structure of 4,4'-diethyl-3,3',5',6-tetramethyl-5-formylpyrroethane **11**. A similar removal of the BF_2 ligand was brought about in the reduction of the aldehyde **4** with sodium borohydride to give the ethane **11**.

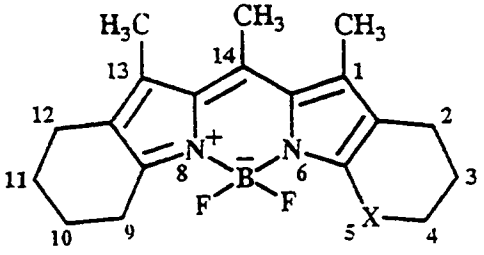
Treatment with lead tetraacetate converted the P–BF_2 complex dye **1** to 1,7,8-trimethyl-2,6-diethyl-3,5-diacetoxymethylpyrromethene–BF_2 complex **12**, a structure confirmed by X-ray crystallographic analysis (Figure 2) [10]. Attempts to hydrolyze the diacetate **12** to the glycol **13** by treatment with either hydrochloric acid, boron trifluoride etherate [15], or alcoholic sodium hydroxide led to intractable mixtures.



1 V = W = Y = Z = CH_3, X = C_2H_5
2 V = W = Y = Z = CH_3, X = H
3 V = H, W = X = Y = Z = CH_3
4 V = W = Z = CH_3, X = C_2H_5
Y = CHO

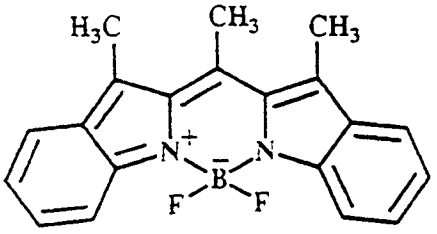
10 V = W = Z = CH_3, X = C_2H_5
Y = CH_2OH
12 V = W = CH_3, X = C_2H_5
Y = Z = CH_2OCOCH_3
13 V = W = CH_3, X = C_2H_5
Y = Z = CH_2OH



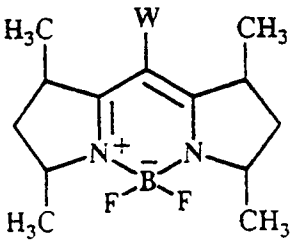


5 X = CH_2
6 X = CO

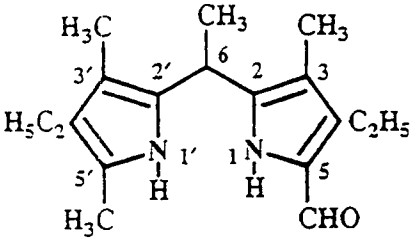
FIGURE 1 Ortep Plot 4



7



8 W = CH_3
9 W = CHO



11

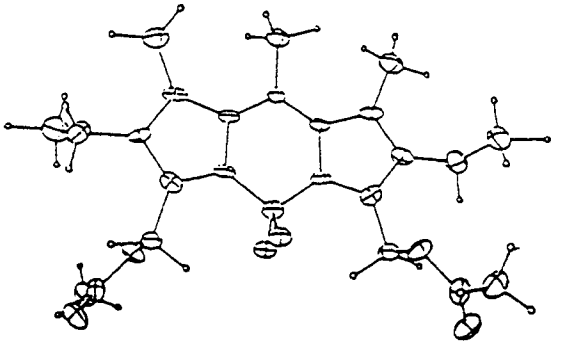


FIGURE 2 Ortep Plot 12

EXPERIMENTAL

Spectral data were obtained from the following instruments: Varian EM 360A, Varian Gemini–300, Varian Unity 400 NMR, Hewlett–Packard 5985 (70 eV) GC–MS, Cary 17 UV, Perkin–Elmer LS–5B Luminescence Spectrometer, Perkin–Elmer 1600 FT–IR, Perkin–Elmer Lambda 6 (UV/VIS) Spectrometer, and a Phase–R DL–1100 dye laser with a DL–5Y coaxial flashlamp. Elemental analyses were obtained from Galbraith Laboratories, Inc., Knoxville, TN, and Midwest Micro Lab, Indianapolis, IN. Melting points were obtained from a Laboratory Devices Mel–Temp II and were uncorrected. IR data supported assigned structures; selected peaks are reported here. Directions for the preparation of P–BF_2 complexes **1**, **2**, and **3** were followed [1]. Low fluorescence quantum yields, $\Phi \sim 0.1$, were observed for dyes **4**, **6**, **8**, and **9**.

2,6-Diethyl-3-formyl-1,5,7,8-tetramethylpyrromethene–BF_2 Complex **4**

Over a period of 2 hours, DDQ (1.82 g, 8.0 mmol) in tetrahydrofuran (16 mL) was added at 0°C under nitrogen to a solution of 2,6-diethyl-1,3,5,7,8-pentamethylpyrromethene–BF_2 complex **1** (0.64 g, 2.0 mmol) in aqueous tetrahydrofuran (99%, 100 mL). After the solution had been stored at 0°C for 4 hours and stirred at room temperature for 24 hours, removal of solvent left a residue which was extracted with methylene chloride, and the filtered solution was chromatographed on a silica gel column. Elution with a mixture of toluene and hexane gave an unidentified purple oil (0.04 g); further elution with a mixture (9/1) of methylene chloride and ethyl acetate gave 2,6-diethyl-3-formyl-1,5,7,8-tetramethylpyrromethene–BF_2 complex **4** as an orange solid (0.53 g, 80%), mp 196–197°C (dec). IR (KBr): ν 1670 (CO). ^{1}H NMR ($CDCl_3$): δ 10.35 (s, 1H, CHO), 2.83 (q, 2H, CH_2), 2.72 (s, 3H, CH_3), 2.62 (s, 3H, CH_3), 2.46 (q, 2H, CH_2), 2.41 (s, 3H, CH_3), 2.34 (s, 3H, CH_3), 1.10 (t, 3H, CH_3), 1.09 (t, 3H, CH_3); ^{13}C NMR ($CDCl_3$): δ 186.00, 163.99, 142.18, 142.00, 138.83, 137.45, 136.69, 136.51, 133.38, 132.02, 17.78, 17.63, 17.17, 14.95, 14.63, 14.31, 13.45, 13.11. EI–MS m/z (%): 332 (M^+, 19). UV (ethanol) λ_{max} (log ϵ) 505 nm (4.03); λ_f (ethanol) 547 nm. Anal. calcd for $C_{18}H_{23}N_2OBF_2$: C, 65.08; H, 6.98; N, 8.43. Found: C, 65.06; H, 7.05; N, 8.19.

1,7,8-Trimethyl-2,6-diethyl-3,5-diacetoxymethylpyrromethene–BF_2 Complex **12**

A procedure for a lead tetraacetate oxidation of a dimethyldipyrryl ketone [16] was adapted. A mixture of 2,6-diethyl-1,3,5,7,8-pentamethylpyrromethene–BF_2 complex **1** (0.32 g, 1.0 mmol), lead tetraacetate (0.89 g, 1.9 mmol), and acetic acid (25 mL) was stirred at 25°C for 12 hours, and then poured into water (200 mL). The solid was collected by filtration, dried, and purified by silica gel

column chromatography (75 g, 230–400 mesh, 60 Å, chloroform) to give 1,7,8-trimethyl-2,6-diethyl-3,5-diacetoxymethylpyrromethene–BF_2 complex **12** (0.25 g, 55%) as an orange solid, mp 187–189°C (dec). IR (KBr): ν 1749, 1552, 1458, 1338, 1128, 864. ^{1}H NMR ($CDCl_3$): δ 5.34 (s, 4H), 2.70 (s, 3H), 2.47 (q, 4H), 2.38 (s, 6H), 2.10 (s, 6H), 1.07 (t, 6H). ^{13}C NMR ($CDCl_3$): δ 170.02, 147.61, 145.12, 137.98, 134.33, 132.67, 56.97, 20.62, 17.46, 16.79, 14.97, 13.97. UV (ethanol) λ_{max} 517 nm, log ϵ 4.71, fluorescence (ethanol) λ_{max} 538 nm, Φ = 0.62 (acridine orange reference standard). Lasing (ethanol) λ_{las} 555–570 nm. Anal. calcd for $C_{22}H_{29}N_2BF_2O_4$: C, 60.82; H, 6.68; N, 6.45. Found: C, 60.69; H, 6.76; N, 6.31.

A less efficient oxidation (29% yield) was brought about by the substitution of chloroform for acetic acid.

4,4'-Diethyl-3,3',5',6-tetramethyl-5-formylpyrroethane **11**

Sodium borohydride (0.02 g, 0.53 mmol) was added to a stirred solution of 2,6-diethyl-3-formyl-1,5,7,8-tetramethylpyrromethene–BF_2 complex **4** (0.17 g, 0.5 mmol) in slightly moist tetrahydrofuran (25 mL). After stirring (2 hours, 25°C), aqueous monosodium phosphate (4%, 45 mL) was added and the mixture was extracted with ether (3 × 35 mL). The combined ether extracts were dried ($MgSO_4$) and evaporated to leave a solid residue (0.16 g). Purification by chromatography [silica gel, methylene chloride/ethyl acetate (9/1)] gave the pyrroethane **11** as a hemihydrate and isolated as an off-white solid (0.07 g, 45%), mp 190–191°C (dec). IR (KBr): ν 3315 (NH), 1614 (CO). ^{1}H NMR ($CDCl_3$): δ 10.62 (br, 1H, NH), 9.40 (s, 1H, CHO), 9.26 (br, 1H, NH), 4.32 (q, 1H, CH), 2.68 (q, 2H, CH_2), 2.33 (q, 2H, CH_2), 2.11 (s, 3H, CH_3), 2.01 (s, 3H, CH_3), 1.99 (s, 3H, CH_3), 1.60 (d, 3H, CH_3), 1.18 (t, 3H, CH_3), 1.04 (t, 3H, CH_3); ^{13}C NMR ($CDCl_3$): δ 175.69, 145.01, 141.26, 127.80, 125.78, 122.27, 120.46, 117.03, 112.98, 28.53, 20.20, 18.18, 17.77, 16.99, 16.05, 11.51, 9.64, 8.94. EI–MS m/z (%): 286 (M^+, 100). Anal. calcd for $C_{18}H_{26}N_2O \cdot 0.5H_2O$: C, 73.18; H, 9.21; N, 9.48. Found: C, 73.47; H, 8.80; N, 9.36.

Hydrogenation of the Aldehyde **4**

2,6-Diethyl-3-formyl-1,5,7,8-tetramethylpyrromethene–BF_2 complex **4** (0.318 g, 0.96 mmol) in ethanol (125 mL) was hydrogenated (48 hours) in the presence of palladium on charcoal (5%, 0.18 g). Filtration and evaporation left a solid residue (0.294 g). Purification by chromatography (silica gel, methylene chloride) gave three fractions:

(a) 2,6-Diethyl-3-hydroxymethyl-1,5,7,8-tetramethylpyrromethene–BF_2 complex **10** as an orange solid (0.11 g, 34%), mp 182–183°C (dec). IR (KBr): ν 3568 (OH). ^{1}H NMR ($CDCl_3$): δ 4.72 (d, 2H, OCH_2), 2.63 (s, 3H, CH_3), 2.44 (m, 13H, CH_2 and CH_3), 1.06 (m, 6H, CH_3); ^{13}C NMR ($CDCl_3$): δ 155.81, 149.14, 141.64, 138.92, 134.05, 133.10, 132.11, 131.58, 55.03, 17.23, 16.99, 15.83, 14.22, 12.66. EI–MS m/z (%): 334 (M^+, 40), 285 (100). UV (ethanol) λ_{max} (log ϵ) 518 nm (4.78); λ_f (ethanol) 537 nm, Φ 0.70 (acridine orange); λ_{las} (ethanol) 547–575 nm. Anal. calcd for $C_{18}H_{25}N_2OBF_2$: C, 64.69; H, 7.54; N, 8.38. Found: C, 64.72; H, 7.58; N, 8.23.

(b) 4,4'-Diethyl-3,3',5',6-tetramethyl-5-formylpyrroethane **11**, 0.02 g (7%); IR and ^{1}H NMR values were identical with the authentic data for an independently prepared sample (above).

(c) 2, 6-Diethyl-1, 3, 5, 7, 8-pentamethylpyrromethene–BF_2 complex **1** (0.06 g, 18%), mp, IR, and ^{1}H NMR identical with authentic data [1].

1,13,14-Trimethyl-2,3,4,5,9,10,11,12-octahydro-5-oxoindomethene–BF_2 Complex **6**

DDQ (1.13 g, 5.0 mmol) was added to a solution of 1,13,14-trimethyl-2,3,4,5,9,10,11,12-octahydroindomethene–BF_2 complex **5** [1] (0.17 g, 0.5 mmol) in benzene (50 mL). The mixture was stirred under nitrogen (25°C, 2 hours), the solvent was removed, the residue was extracted with methylene chloride, and the filtered solution was chromatographed on a silica gel column. Elution with a mixture (10/1) of methylene chloride and ethyl acetate gave the oxoindomethene–BF_2 complex **6** as an orange solid (0.08 g, 47%), mp 212–214°C (dec). IR (KBr): ν 1675 (CO). ^{1}H NMR ($CDCl_3$): δ 3.16 (t, 2H, CH_2), 2.66 (s, 3H, CH_3), 2.59 (m, 4H, CH_2), 2.46 (t, 2H, CH_2), 2.31 (s, 6H, CH_3), 2.06 (m, 2H, CH_2), 1.78 (m, 4H, CH_2); ^{13}C NMR ($CDCl_3$): δ 189.05, 166.85, 141.82, 140.99, 137.78, 137.42, 137.28, 134.37, 132.80, 129.17, 40.52, 25.52, 23.82, 22.19, 22.00, 21.89, 21.77, 17.64, 14.61, 13.97. EI–MS m/z (%): 356 (M^+, 100). UV (ethanol) λ_{max} (log ϵ) 500 nm (4.34); λ_f (ethanol) 531 nm. Anal. calcd for $C_{20}H_{23}N_2OBF_2$: C, 67.43; H, 6.51; N, 7.86. Found: C, 67.48; H, 6.63; N, 7.59.

1,3,5,7,8-Pentamethylhexahydropyrromethene–BF_2 Complex **8**

A previous procedure [17] was modified. The fluorescent 1,3,5,7,8-pentamethylpyrromethene–BF_2 complex **2** (0.6 g, 2.3 mmol) in ethanol (95%, 200 mL) was hydrogenated in the presence of 5% palladium on charcoal for 17 hours at 25°C. Filtration, concentration, and recrystallization (pentane) gave 1,3,5,7,8-pentamethylhexahydropyrromethene–BF_2 complex **8** as a colorless nonfluorescent crystalline solid (0.49 g, 80%), mp 129–130°C [17]. ^{1}H NMR

($CDCl_3$): δ 4.03–4.30 (m, 2H, CH), 2.67–3.30 (m, 2H, CH), 2.40–2.80 (m, 2H, CH_2), 1.77–1.97 (m, 2H, CH_2), 1.23–1.53 (m, 15H, CH_3). EI–MS m/z (%): 268 (M^+, 18), 253 (100). UV (ethanol) λ_{max} (log ϵ) 334 nm (4.28); λ_f (ethanol) 365 nm.

8-Formyl-1,3,5,7-tetramethylhexahydropyrromethene–BF_2 Complex **9**

2,3-Dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) (0.69 g, 3.0 mmol) was added to a solution of 1,3,5,7,8-pentamethylhexahydropyrromethene–BF_2 complex **8** (0.40 g, 1.5 mmol) in benzene (30 mL). The mixture was stirred under nitrogen (25°C, 17 hours), and the solvent was evaporated. The residue was extracted with methylene chloride (3 × 5 mL), and the filtered methylene chloride solution was chromatographed on a silica gel column. Elution with a mixture (1:1) of methylene chloride and ethyl acetate gave 8-formyl-1,3,5,7-tetramethylhexahydropyrromethene–BF_2 complex **9** as a colorless solid (0.29 g, 65%), mp 170–171°C IR (KBr): ν 2765 (CHO), 1656 (CO). ^1H NMR ($CDCl_3$): δ 9.65 (s, 1H, CHO), 4.27–4.43 (t, 2H, CH), 3.69–3.87 (t, 2H, CH), 2.37–2.72 (m, 2H, CH_2), 1.42–1.59 (m, 14H, CH_3 and CH_2); ^{13}C NMR ($CDCl_3$): δ 184.00, 177.06, 59.25, 39.83, 37.10, 30.87, 23.56, 22.12. EI–MS m/z (%): 282 (M^+, 62), 267 (100). UV (ethanol) λ_{max} (log ϵ) 265 nm (4.27); λ_f (ethanol) 355 nm. Anal. calcd for $C_{14}H_{21}N_2OBF_2$: C, 59.60; H, 7.50; N, 9.93. Found: C, 59.70; H, 7.43; N, 9.63.

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