was extracted with diethyl ether (3 × 30 mL). The combined organic layer was dried ($MgSO_4$) and concentrated. The residue was purified by column chromatography on silica gel (petroleum spirit:diethyl ether = 3:1) to give 1.1 g of pure product (86% yield). ^1H NMR (90 MHz, $CDCl_3$): δ 3.03 (d, 2 H, J = 5.25 Hz, CH_2), 3.2 (d, 2 H, J = 4.16 Hz, CH_2), 3.8 (s, 3 H, OMe), 5.78–5.99 (m, 4 H, olefinic protons). ^{13}C NMR (22.5 MHz, $CDCl_3$): δ 29.44 (t), 35.27 (t), 61.19 (q, OMe), 127.39 (d), 127.52 (d), 127.77 (d), 127.96 (d), 164.41 (s, C-7). IR (neat, cm^{-1}): 3024, 2937, 1627, 1424, 1052. HRMS for $C_8H_{11}NO$: calcd 137.0885, found 137.0821. Anal. Calcd for $C_8H_{11}NO$: C, 70.04; H, 8.08; N, 10.21. Found: C, 70.60; H, 8.13; N, 9.88.

1-(*N*-Methoxy-*N*-(trimethylsilyl)amino)cyclohepta-1,3,5-triene (2). This compound is highly sensitive to moisture and was prepared and worked up by the method described below.

Trimethylsilyl trifluoromethanesulfonate (1.22 g, 5.5 mmol) was added cautiously with a syringe to a stirring solution of the *O*-methyloxime 1 (0.69 g, 5 mmol) and triethylamine (0.61 g, 6 mmol) in dried *n*-hexane (15 mL) under dry nitrogen atmosphere. Stirring was continued until two immiscible layers were formed. The lower layer was darkish brown in color. About 500 μL of the upper clear layer was withdrawn from the reaction flask and transferred immediately to a NMR tube dried in a desiccator over P_2O_5 for at least 1 day. The NMR tube was transferred quickly to a vacuum trap, and the organic solvent was removed at reduced pressure with great care to prevent the solution from shooting out of the NMR tube. After all the organic solvent was removed, dry air was admitted to the vacuum trap through an anhydrous $CaCl_2$ guard tube. Deuterated solvent (DMSO-d_6) was added, and the spectra were recorded immediately. MS (m/e): 225 (M^+).

Registry No. 1, 142868-78-8; 2, 142868-79-9; 3, 142868-80-2; 4, 142868-81-3; $MeONH_2$·HCl, 593-56-6; 3,5-cycloheptadien-1-one, 1121-65-9.

Anti-Michael Addition and Fluoride Ion Elimination on β,β-Bis(trifluoromethyl)acrylic Esters. Preparation of α-Perfluoroisopropenyl α-Substituted Acetic Acid Esters[1]

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Received April 14, 1992

In the course of our studies on fluorinated analogues of prenyl derivatives[2] the allylic alcohol **2** was needed. This compound was already described in a patent[3] and by Taguchi.[4] Both reported methods referred to a metal hydride reduction of the readily prepared ethyl β,β-bis(trifluoromethyl)acrylic ester (**1**).[5] In our hands, all attempts to repeat these described procedures failed. With the recommended diisobutylaluminum hydride[6] (DIBALH) in toluene at −70 °C we obtained the perfluoroisopropenyl compound **4a** resulting from an anti-Michael addition[7] of hydride ion followed by a fluoride ion elimination. Two other unidentified fluorinated products were also formed. The inverted polarization of the double bond in β,β-bis(trifluoromethyl)acrylic esters has already been observed by Knunyants in the addition of amines which apparently was not followed by fluoride ion elimination.[8] This unexpected addition–elimination reaction was extended to various nucleophiles, and we report here a convenient route to the new α-perfluoroisopropenyl α-substituted acetic acid esters **4**.[9]

Compound **4a** was obtained as a single product when using the less reactive lithium triethoxyaluminum hydride instead of DIBALH for the reduction. The reaction was conducted in ether at −78 °C, and **4a** was isolated in 77% yield. The sodium salt of diethyl malonate in tetrahydrofuran at 10 °C gave **4b** in 70% yield. Under the same conditions, the sulfide **4c** was obtained with 67% yield from the sodium phenyl sulfide. Organolithium reagents reacted in this fashion as well. For example, methyllithium in tetrahydrofuran at −78 °C gave **4d** in 54% yield. In all cases, the hexafluoro compounds **5a–d** were formed in less than 5% as estimated from the ^{19}F NMR spectra of the crude reaction product, but could not be isolated.[10] With piperidine, only the saturated compound **5e** was isolated as previously reported by Knunyants.[8] However careful monitoring during the course of the reaction by ^{19}F NMR showed the formation of **4e** as major product at the beginning of the condensation. We noticed that increasing the reaction time led only to **5e** which could be considered as the thermodynamic product. All attempts to isolate **4e** failed.

These observations allow us to postulate the formation of the carbanionic intermediate **3** which, in the absence of a proton source, will eliminate fluoride ion to form **4**. In the case of the amine, a piperidinium hydrofluoride salt was formed and the fluoride ion became nucleophilic enough to add to the fluorinated double bond[11] of the kinetic product **4e** to give **5e**. The analogous tetrabutylammonium fluoride addition to **4** leading to the bistrifluoromethyl compounds **5**, which was followed by ^{19}F NMR, was in agreement with this mechanism.

This anti-Michael addition requires two trihalogenomethyl electron-withdrawing groups as Walborsky[12] showed that γ,γ,γ-trifluorocrotonate reacted in the same manner as its nonfluorinated analog. In the competition between a single trifluoromethyl group and carboxyl group, the latter one determined the regioselectivity of the addition.

A similar anti-Michael addition of nucleophiles followed by halide ion elimination has been observed with hindered γ-bromo α,β-unsaturated esters.[13] An allylic displacement reaction was also observed when treating highly halogenated olefins with lithium aluminum hydride since terminal perfluoromethylene olefins were formed.[14] Recently, α-perfluoroisopropenyl ketones have been obtained from

(1) Presented in part at the XIIIth International Symposium on Fluorine Chemistry, Bochum (FRG), September 1991.

(2) 1-Bromo-3-(trifluoromethyl)but-2-ene: synthesis and electrophilic reactivity. Martin, V.; Molines, H.; Wakselman, C. *J. Fluorine. Chem.*, in press.

(3) Noyori, R.; Morisawa, Y.; Maeda, T.; Yasuda, A.; Uchida, K. JP 02 56,462 [90 56,462]; *Chem. Abstr.* **1990**, *113*, 59620g.

(4) Taguchi, T.; Hosada, A.; Torisawa, Y.; Shimazaki, A.; Kobayashi, Y.; Tsushima, K. *Chem. Pharm. Bull.* **1985**, *33*, 4085.

(5) Abele, H.; Haas, A.; Lieb, M. *Chem. Ber.* **1986**, *119*, 3502.

(6) We thank Professor Takeo Taguchi for the personnal communication giving us a detailed experimental procedure.

(7) For anti-Michael additions see; Klumpp, G. W.; Mierop, A. J. C.; Vrielink, J. J.; Brugman, A.; Schakel, M. *J. Am. Chem. Soc.* **1985**, *107*, 6740 and references cited therein.

(8) Knunyants, I. L.; Cheburkov, Yu. A. *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk* **1960**, 2162.

(9) Only the acid of **4a** has yet been obtained as a mixture with three other fluorinated compounds, by treatment of perfluoro-2,2-dimethylbutadiene with water. Kaz'mina, N. B.; Krasnikova, G. S.; Lur'e, E. P.; Mysov, E. I.; Knunyants, I. L. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1975**, 2525.

(10) Compound **5a** was isolated by preparative GC (90 °C): ^1H NMR (200 MHz) 1.35 (t, 3 H, $^3J_{HH}$ = 7.1 Hz), 2.8 (d, 2 H, $^3J_{HH}$ = 6.04 Hz), 3.78 (septuplet × d, 1 H, $^3J_{HH}$ = 6.04 Hz, $^3J_{HF}$ = 8.5 Hz), 4.25 (q, 2 H, $^3J_{HH}$ = 7.1 Hz); ^{19}F NMR −68.63 (d, $^3J_{HF}$ = 8.5 Hz).

(11) Molines, H.; Wakselman, C. *J. Fluorine Chem.* **1984**, *25*, 447.

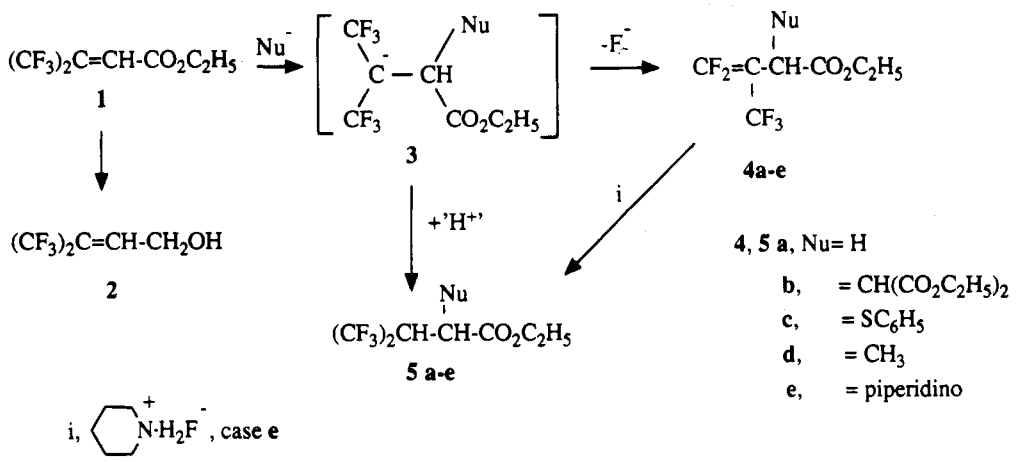
(12) Walborsky, H. M.; Schwarz, M. *J. Am. Chem. Soc.* **1953**, *75*, 3241.

(13) Roux-Schmitt, M. C.; Sevin, A.; Seyden-Penne, J. *Bull. Soc. Chim. Fr.* **1990**, *127*, 857.

(14) Kaufman, M. H.; Braun, J. D.; Shdo, J. G. *J. Org. Chem.* **1967**, *32*, 2749.

0022-3263/92/1957-5530$03.00/0

Scheme I



β,β-bis(trifluoromethyl) α,β-unsaturated ketones. However, different reactions were involved in this process so the mechanism must be completely different.[15]

Experimental Section

General Methods. ^1H NMR spectra were recorded in $CDCl_3$. ^{19}F NMR spectra were recorded at 282.38 MHz. The ^{19}F NMR monitoring of the reaction with piperidine was performed at 56.4 MHz. ^{19}F NMR chemical shifts are reported in parts per million (ppm), negative upfield from trichlorofluoromethane. IR spectra were obtained in CCl_4. Preparative GC was performed with a 2.5-m SE-30 column. Ether was diethyl ether. Dry ether and THF were distilled from sodium just before use. Brine is a saturated aqueous NaCl solution. Hexafluoroacetone and methyllithium in ether were purchased from Aldrich-Chimie. All reactions were conducted under inert atmosphere (argon) in a flame-dried three-necked flask equipped with a funnel, a thermometer, and a condenser fitted with a silica gel-filled drying tube.

Ethyl 4,4-Difluoro-3-(trifluoromethyl)but-3-enoate (4a). A solution of lithium triethoxyaluminum hydride[16] (29.5 mL) was added by portions (3 mL) to a solution of ethyl β,β-bis(trifluoromethyl)acrylate (5 g, 21 mmol) in dry ether (40 mL) cooled to −78 °C. After complete addition, the mixture was stirred at this temperature, and then methanol (1.1 mL) was added. The mixture was allowed to warm to −10 °C, and then water (1.1 mL), 15% aqueous NaOH (1.1 mL), and again water (3.3 mL) were added successively. The hydrolysis was complete when a white precipitate appeared. The mixture was filtered, and the aluminum salt was washed with ether (2 × 30 mL). The ethereal solution was dried (Na_2SO_4). The solvent was distilled off and the residue distilled under reduced pressure (185 mmHg) to give **4a** (3.56 g, 16.3 mmol, 77%): bp 80 °C (185 mmHg); ^1H NMR (300 MHz) 1.33 (t, 3 H, $^3J_{HH}$ = 7.15 Hz), 3.22 (dd, 2 H, $^4J_{HF}$ = 2.05 and 2.1 Hz), 4.25 (q, 2 H, $^3J_{HH}$ = 7.15 Hz); ^{19}F NMR −61.6 (dd, 3 F, $^4J_{FF}$ = 20.6 and 11.5 Hz), −75.16 (qtd, 1 F, $^2J_{FF}$ = 14.8 Hz, $^4J_{FF}$ = 20.6 Hz, $^4J_{HF}$ = 2.05 Hz), −78 (qtd, 1 F, $^2J_{FF}$ = 14.8 Hz, $^4J_{FF}$ = 11.5 Hz, $^4J_{HF}$ = 2.1 Hz); IR 2970–2850, 1745, 1365, 1325, 1285, 1250, 1190, 1170, 1130, 1020 cm^{-1}. Anal. Calcd for $C_7H_7F_5O_2$ (218.121): C, 38.55; H, 3.235. Found: C, 38.56; H, 3.27.

Ethyl 2,2-[Bis(ethoxycarbonyl)methyl]-4,4-difluoro-3-(trifluoromethyl)but-3-enoate (4b). To a sodium hydride suspension (60% in mineral oil 0.726 g, 18 mmol; washed with pentane (3 × 5 mL)) in dry THF (40 mL), under an inert atmosphere, was added diethyl malonate (2.73 g, 17 mmol) dropwise at rt. This mixture was allowed to decant, and the liquid phase was collected via a syringe and added dropwise to a solution of ethyl β,β-bis(trifluoromethyl)acrylate (3 g, 12.7 mmol) in dry THF (30 mL) at 10 °C. The mixture was stirred at this temperature for 30 min. The reaction was then quenched with 2% HCl (50 mL) and extracted with ether (2 × 80 mL). The organic solution was dried (Na_2SO_4). The solvents were evaporated (20 mmHg), and the residue was bulb-to-bulb distilled (0.1 mmHg). The distillate was then redistilled under reduced pressure (18 mmHg) to give **4b** (3.32 g, 8.83 mmol, 70%): bp 130 °C (18 mmHg); ^1H NMR (300 MHz) 1.31 (t, 6 H, $^3J_{HH}$ = 7.05 Hz), 1.34 (t, 3 H, $^3J_{HH}$ = 7.05 Hz), 4.06 (d, 1 H, $^3J_{HH}$ = 11.7 Hz), 4.27 (m, 7 H); ^{19}F NMR −58.85 (dd, 3 F, $^4J_{FF}$ = 10.3 and 16.5 Hz), −71.65 (qd, 1 F, $^2J_{FF}$ = 5.13 Hz, $^4J_{FF}$ = 16.5 Hz), −74.65 (m, 1 F); IR 2960, 2920, 2880, 2850, 1740, 1455, 1435, 1350, 1270, 1170, 1140, 1090, 1010 cm^{-1}. Anal. Calcd for $C_{14}H_{17}F_5O_6$ (326.274): C, 44.69; H, 4.55. Found: C, 44.75; H, 4.65.

Ethyl 4,4-Difluoro-3-(trifluoromethyl)-2-(phenylthio)-but-3-enoate (4c). This compound was prepared by the same procedure as for **4b** using the following amounts of materials: 60% sodium hydride in mineral oil dispersion (0.76 g, 19 mmol), thiophenol (2.1 g, 19 mmol), dry THF (70 mL), and ethyl β,β-bis(trifluoromethyl)acrylate (3 g, 12.7 mmol) to give after distillation **4c** (2.78 g, 8.52 mmol, 67%): bp 145 °C (1 mmHg); ^1H NMR (200 MHz) 1.21 (t, 3 H, $^3J_{HH}$ = 7.1 Hz), 4.19 (2 q, 2 H, $^3J_{HH}$ = 7.1 Hz), 4.57 (s, 1 H), 7.26–7.52 (m, 5 H); ^{19}F NMR −58.66 (dd, 3 F, $^4J_{FF}$ = 9.6 and 14.8 Hz), −73.64 (qd, 1 F, $^2J_{FF}$ = 9.6 Hz, $^4J_{FF}$ = 14.8 Hz), −77.66 (qd, 1 F, $^2J_{FF}$ = 9.6 Hz, $^4J_{FF}$ = 9.6 Hz); IR 3050, 2970, 2920, 2890, 2860, 1750, 1470, 1430, 1350, 1270, 1220, 1180, 1140, 1090, 1020, 1000 cm^{-1}. Anal. Calcd for $C_{13}H_{11}F_5O_2S$ (326.283): C, 47.85; H, 3.398. Found: C, 47.92; H, 3.41.

Ethyl 4,4-Difluoro-2-methyl-3-(trifluoromethyl)but-3-enoate (4d). A solution of methyllithium in ether (1.6 M, 5.31 mL, 8.5 mmol) was added dropwise, under an inert atmosphere, to ethyl β,β-bis(trifluoromethyl)acrylate (2 g, 8.5 mmol) in dry THF (10 mL) at −78 °C. The mixture was stirred at this temperature for 30 min, and then water (10 mL) and 5% HCl (5 mL) were added. The mixture was allowed to warm to room temperature and was then extracted with ether (3 × 30 mL). The organic layer was washed with brine and dried (Na_2SO_4). The solvents were distilled off. The residue was distilled under reduced pressure to give **4d** (1.06 g, 4.56 mmol, 54%): bp 80 °C (185 mmHg); ^1H NMR (200 MHz) 1.19 (t, 3 H, $^3J_{HH}$ = 7.12 Hz), 1.36 (d, 3 H, $^3J_{HH}$ = 7.35 Hz), 3.33 (q, 1 H, $^3J_{HH}$ = 7.35 Hz), 4.13 (2 q, 2 H, $^3J_{HH}$ = 7.12 Hz); ^{19}F NMR −59.75 (dd, 3 F, $^4J_{FF}$ = 10.3 and 19.75 Hz), −75.3 (qd, 1 F, $^2J_{FF}$ = 17.5 Hz, $^4J_{FF}$ = 19.75 Hz), −79.23 (broad qd, 1 F, $^2J_{FF}$ = 17.5 Hz, $^4J_{FF}$ = 10.3 Hz); IR 2980, 2930, 2900, 2860, 1740, 1455, 1440, 1375, 1350, 1320, 1280, 1260, 1215, 1175, 1150, 1125, 1090, 1050, 1020 cm^{-1}. Anal. Calcd for $C_8H_9F_5O_2$ (232.148): C, 41.39; H, 3.91. Found: C, 41.30; H, 3.99.

Ethyl 4,4,4-Trifluoro-3-(trifluoromethyl)-2-piperidinobutanoate (5e). Piperidine (2.125 g, 25 mmol) was added dropwise to ethyl β,β-bis(trifluoromethyl)acrylate (3 g, 12.7 mmol) in dry ether (25 mL) at 0 °C. After the addition, the mixture was stirred at rt. The monitoring of the reaction by ^{19}F NMR (at 56.4 MHz) made it possible to observe the formation of ethyl 4,4-difluoro-3-(trifluoromethyl)-2-(piperidino)but-3-enoate (**4e**) (−63 (dd, 3 F), −79.3 (m, 1 F), −83.3 (m, 1 F)) in the mixture with **5e** (−60.3

(15) Burger, K.; Helmreich, B. *J. Chem. Soc., Chem. Commun.* **1992**, 348.

(16) The lithium triethoxyaluminum hydride solution was prepared as follows: in a flame-dried two-necked flask under inert atmosphere (argon) a solution of ethanol (2.92 g, 63 mmol) in dry ether (50 mL) was slowly added (1 h) to a suspension of $LiAlH_4$ (3.19 g, 84 mmol) in dry ether (80 mL) cooled to 0 °C; the resulting mixture was then stirred for 1 h and then could be used.

(q, 3 F), −63.5 (q, 3 F)). The ratio evolved with time, and after 2 h no more **4e** was detected on the ^{19}F NMR spectrum. Then, 5% HCl (25 mL) was added. The mixture was extracted with ether (2 × 20 mL). The organic layer was dried ($MgSO_4$). The solvent was evaporated (20 mmHg), and the residue was bulb-to-bulb distilled (15 mmHg) to give an oil which was crystallized from ethanol (95%) affording **5e** as white crystals (3.01 g, 10.64 mmol, 74%): mp 25.4 °C (lit.[9] mp 26.5–27.5 °C); ^1H NMR (200 MHz) 1.24 (t, 3 H, $^3J_{HH}$ = 7.13 Hz), 1.27–1.36 (m, 2 H), 1.43–1.52 (m, 4 H), 2.24 (m, 2 H), 2.59 (m, 2 H), 3.51 (d, 1 H, $^3J_{HH}$ = 11.03 Hz), 3.70 (d × septuplet, 1 H, $^3J_{HH}$ = 11.03 Hz, $^3J_{HF}$ = 7.82 Hz), 4.17 (q, 2 H, $^3J_{HH}$ = 7.13 Hz); ^{19}F NMR −60.3 (q, 3 F, $^3J_{HF}$ = 7.82 Hz), −63.5 (q, 3 F, $^3J_{HF}$ = 7.82 Hz).

Registry No. **1**, 1513-60-6; **4a**, 58064-40-7; **4b**, 143063-57-4; **4c**, 143063-58-5; **4d**, 143063-59-6; **4e**, 143063-60-9; **5e**, 1763-89-9; $CH_2(CO_2Et)_2$, 105-53-3; PhSH, 108-98-5; piperidine, 110-89-4.

Heterocyclic *o*-Quinodimethanes in Synthesis: A Diels–Alder Approach to Xanthene-Derived Heterocycles

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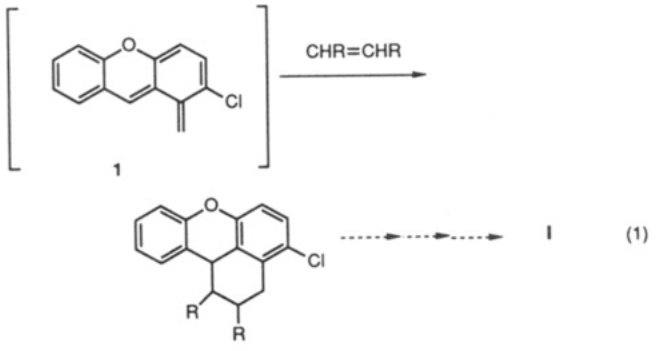
The [4 + 2] cycloaddition strategy has found wide application for six-membered-ring construction in complex systems. Since *o*-quinodimethanes are highly reactive diene components, they have been very effective in this arena for the assembly of polycyclic aromatic compounds.[1] During studies aimed at discovering novel antipsychotic agents,[2] we sought to employ this approach as a general synthetic entry into the ring system exemplified by formula **I**. Thus, *o*-quinodimethane **1** emerged as an attractive intermediate (eq 1). On the basis of literature precedent



for the use of *o*-xylyl dihalides as *o*-quinodimethane precursors,[1] we decided to evaluate the suitability of **2** as a precursor to **1**. Herein, we describe the in situ generation of **1** and trapping experiments with various dienophiles en route to polycyclic molecules.

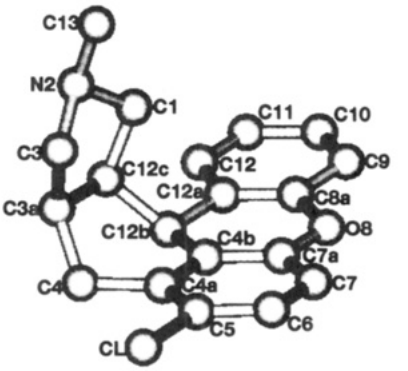


Figure 1. SYBYL drawing of the hydrochloride salt of **14**.

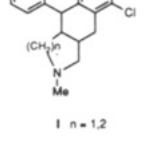
Results and Discussion

Synthesis of **2** is outlined in Scheme I. En route to **2**, a novel approach regiospecifically furnished the previously unreported xanthonecarboxylic acid **3**. Thus, Grignard addition of 2-methoxyphenylmagnesium bromide to 3,6-dichlorophthalic anhydride[3] (**4**) gave keto acid **5**. Demethylation of **5**, followed by cyclization of the resulting phenolic keto acid **6**, according to the conditions of v. dem Knesebeck,[4] furnished xanthone acid **3** in 70% overall yield. Next, we needed to transform the carbonyl and carboxyl functionalities of **3** into benzhydryl and benzyl chlorides, respectively. Since treatment of **3** with BH_3–THF resulted in over-reduction to alcohol **8**,[5] we accessed **7** via $LiAlH_4$ reduction of methyl ester **9**. Treatment of **7** with excess thionyl chloride gave the desired *o*-quinodimethane precursor **2** as a stable, crystalline solid. Scheme II highlights the reactivity of **2** toward various dienophiles.

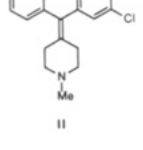
Addition of NaI[6] to a DMF solution of **2** and excess *N*-methylmaleimide at 73 °C gave a major product in 45% yield whose 360-MHz ^1H NMR spectrum was generally consistent with the expected pentacyclic adduct. Definitive proof of structure was obtained through X-ray analysis of **14**, a reduced derivative of **10a** (vide infra, Figure 1). A doublet at 4.33 ppm (J = 5.69 Hz) for H_{12b} and a doublet of doublets at 3.71 ppm (J = 5.66, 9.06 Hz) for H_{12c} led us to assign structure **10a** to the product. This is reflective of Diels–Alder addition in the expected endo mode. An attempt to effect thermal isomerization of **10a** to the exo adduct **10b** (sealed NMR tube, $CDCl_3$, 110 °C) was unsuccessful, while base treatment (DBU) gave a mixture of starting material and aromatized product, **11**.[7] The ap-

(1) For a recent review of *o*-quinodimethanes, see: Charlton, J. L.; Alauddin, M. N. *Tetrahedron* **1987**, *43*, 2873.

(2) For a review of the pharmacology and chemistry of antipsychotic agents see: (a) Kaiser, C.; Setler, P. E. In *Burger's Medicinal Chemistry*, 4th ed.; Wolff, M. E., Ed.; John Wiley & Sons: New York, 1981; Part III, Chapter 56, p 859. Structures embodied by formula **I** were patterned after clopipazan (**II**), a compound which has high affinity for the serotonin (5-HT_2) and dopamine (D_2) receptors; see: (b) Middlemiss, D. N.; Hibert, M.; Fozard, J. R. *Annu. Rep. Med. Chem.* **1986**, *21*, 41.



I n = 1,2



II

(3) Villiger, V. *Chem. Ber.* **1909**, *42*, 3529. For a significant improvement of the preparation of 3,6-dichlorophthalic anhydride, see Experimental Section.

(4) v. dem Knesebeck, A. M.; Ullmann, F. *Chem. Ber.* **1922**, *55*, 306. These authors prepared 2-chloro-7-methylxanthene-1-carboxylic acid by a Friedel–Crafts acylation of *p*-cresol with 3,6-dichlorophthalic anhydride. In this case, regiospecificity clearly is not a problem since the para position is blocked. In our hands, Friedel–Crafts acylation of phenol with **4** gave a separable mixture of (2′-hydroxybenzoyl)- and (4′-hydroxybenzoyl)benzoic acids.

(5) Wechter, W. J. *J. Org. Chem.* **1963**, *28*, 2935. Compound **8** was characterized by NMR only: ^1H NMR (DMSO-d_6) δ 4.43 (s, 2 H), 4.66 (d, 2 H, J = 5.37 Hz), 5.14 (t, 1 H, J = 5.37 Hz), 7.03–7.32 (m, 6 H); mp = 110–111.5 °C (CH_3CN).

(6) Cava, M. P.; Deana, A. A.; Muth, K. *J. Am. Chem. Soc.* **1959**, *81*, 6458.

(7) This reaction was not run with rigorous exclusion of air. Compound **11** was identical by TLC and ^1H NMR to material alternatively produced by DDQ oxidation of **10a** (Experimental Section).

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