OCTAFLUORO[2.2]PARACYCLOPHANE (AF4) QUINONE

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Dedicated to Professor Oldřich Paleta on the occasion of his 70th birthday in recognition of his outstanding contributions to the field of organofluorine chemistry.

Octafluoro[2.2]paracyclophane (AF4) has been oxidized by treatment with HIO_3 in CF_3CO_2H to form the corresponding *p*-quinone along with a unique triketone. This quinone undergoes reduction to the respective hydroquinone as well as a Diels–Alder reaction with 1,3-cyclohexadiene. Its reduction potential was obtained by cyclic voltammetry and is discussed in the context of other quinones.

Keywords: Cyclophane; Fluorinated compounds; Oxidations; [2.2]Paracyclophane; Quinones; Cyclic voltammetry; UV spectra; Diels–Alder reaction.

In our extensive studies of the chemical behavior of 1,1,2,2,9,9,10,10-octafluoro[2.2]paracyclophane (AF4), the iodo derivative has proved particularly useful for a number of reactions. First, it was used in coupling and dimerization reactions using copper catalysis[1,2]. Subsequently it was found to serve as a useful precursor of arynes[3], and lastly it was found to be an excellent substrate for a number of SRN1 reactions[4].

The iodo AF4 could be synthesized via a straightforward three-step sequence involving nitration, reduction to the amine and iodination via the diazonium species[1]. Nevertheless, in a long shot attempt to make the synthesis of the iodo compound more efficient, a direct synthesis was attempted using conditions ($I_2/HIO_3/H_2SO_4$) that are well known to give rise to aromatic iodination for electron rich aromatics[5,6]. The benzene rings of AF4 are, of course, not electron rich – quite the contrary.

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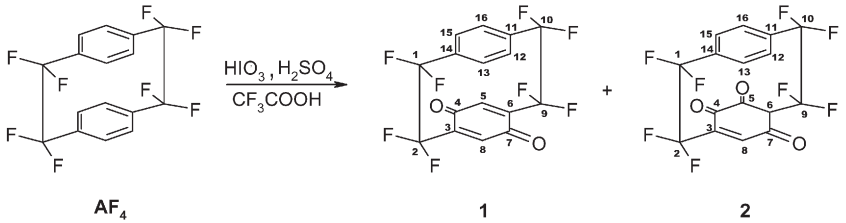
RESULTS AND DISCUSSION

In the event, when AF4 was allowed to react under these conditions, instead of undergoing iodination, it underwent oxidation to form its quinone **1** as the major product (Scheme 1), which was readily characterized by its proton, fluorine and carbon NMR spectra, its mass spectrum, its elemental analysis, and its chemical behavior. The similarities in specific ^{13}C NMR chemical shifts between the AF4-quinone **1** and 2,5-bis(trifluoromethyl)-1,4-benzoquinone[7] are clearly consistent with the assignment of the quinone structure to **1**. That is the chemical shift of the carbonyl carbon is 181.0 in the former and 180.6 in the latter, and the chemical shift of the carbon bearing the CF_2 or CF_3 group is 134.6 in the former (C6) and 134.2 in the latter (C2).



SCHEME 1

A second product, which contained one additional oxygen according to its elemental analysis, was identified by NMR as triketone **2** (Scheme 1 and Table I).

The proton spectrum of compound **2** displayed two AB systems, corresponding to H12–H13 and H15–H16. The carbons in these positions were identified in the gHMQC spectrum. Long-range ^{1}H-^{13}C couplings, seen in the gHMBC spectrum, between the protons and carbons in positions 12 and 16 on one hand, and in positions 13 and 15 on the other, revealed their *meta* relationship. The quaternary carbons on the *para*-phenylene moiety were identified by their couplings with the protons *meta* to them, i.e. C11 with H13 and H15, and C14 with H12 and H16. The benzylic carbons in the *para*-xylylene moiety display cross-peaks with the *ortho* protons (C10 with H12, H16 and C1 with H13, H15) and in the ^{13}C dimension are triplets of triplets (275 and 39 Hz) because of the couplings of these carbons with the four fluorines of the perfluoroethylene chain. The eight non-equivalent fluorines of the ^{19}F spectrum were assigned to two tetrafluoro-

ethylene moieties based on the cross-peaks seen in the ^{19}F-^{19}F TOCSY spectrum, i.e. of the four fluorines in the same coupling network, –120.6, –114.8, –109.7 and –97.0, the first two and the last two are geminal because they couple with a large coupling constant, 250 and 272 Hz, correspondingly. Strong nOe's between –120.6 and 6.80 and between –120.4 and 6.49

TABLE I
^{1}H, ^{13}C and ^{19}F chemical shifts of compound **2**, in benzene-d_6 at 25 °C

Position no.	$\delta^{13}C$ ppm	$\delta^{1}H$ or $\delta^{19}F$ ppm	Position no. of the protons which display cross-peaks in the gHMBC spectrum with the carbon in this position
1	115.6	–121.5, –120.4	13, 15
2	114.9	–103.3, –122.1	8
3	146.7[a]	–	–
4	188.7[b]	–	6, 8
5	nm[c]	–	–
6	52.9	2.23	8
7	185.8	–	6, 8
8	147.1	5.50	–
9	nm[d]	–109.7, –97.0	–
10	114.4	–120.6, –114.8	12, 16
11	131.4	–	13, 15
12	127.4	6.80	16
13	129.9	6.49	15
14	133.9	–	13, 15
15	131.4	7.03	13
16	133.3	6.97	12

[a] Value in DMSO-d_6. The ^{13}C spectrum with ^{1}H broadband decoupling was taken in DMSO-d_6, in which compound **2** is more soluble. In this solvent there are four triplets of ca. 25 Hz at 147.6 (C3), 134.2 (C14), 131.4 (C11) and 54.1 ppm (C6). The same spectrum displays two signals for the carbonyl carbons at 191.5 and 188.0 ppm. [b] Carbonyls in positions 4 and 7 are interchangeable, since they display cross-peaks with the same protons. These assignments are based on chemical shifts. [c] Only two carbonyls showed up in the gHMBC spectrum, both with cross-peaks to H6 and H8, therefore the missing one was assigned to C5, for which a cross-peak to H8 is not expected. [d] No couplings with protons are expected for this carbon, nor has its signal been seen in the ^{13}C spectrum with broadband ^{1}H decoupling in DMSO-d_6, because of the splitting of this signal by the couplings with fluorines.

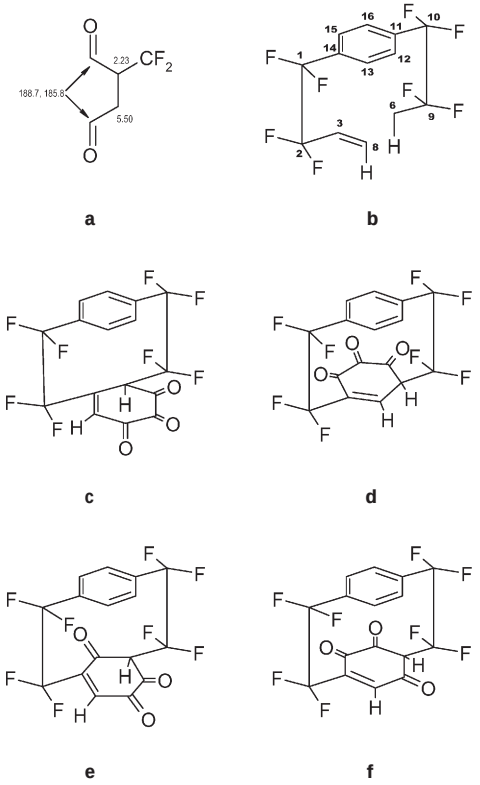
were seen in the HOESY spectrum, and they were the basis for the assignment of the corresponding difluoromethylene groups. The ^{13}C spectrum with broad band ^{1}H decoupling displays four triplets of ca. 25 Hz, corresponding to the carbons linked to the tetrafluoroethylene chains. Two of them have already been assigned as C11 and C14. Of the remaining two, 52.9 and 146.7, the former is protonated and its proton at 2.23 couples with the fluorine at –97.0 with a coupling constant of 16.8 Hz, indicative of their vicinal relationship, therefore the carbons at 52.9 and 146.7 were assigned to positions 6 and 3 correspondingly. Another carbon at 114.9, with the chemical shift and the triplet of triplets pattern of a CF_2, exhibits a cross-peak with proton 5.50 in the gHMBC spectrum. There are two possible assignments for 114.9, C2 and C9. Assuming that 114.9 is C9, then protons 2.23 and 5.50 become vicinal. They both couple with two carbonyl carbons, at 188.7 and 185.8, which have to be adjacent to their carbons, like in structure **2a** of Scheme 2. This is not compatible with the chemical shift, 147.1, of the carbon bearing the proton at 5.50 which cannot be a proton-bearing sp^2 carbon in this arrangement; therefore 114.9 is C2. Up to this point we have demonstrated the partial structure **2b** in Scheme 2, to which we have to add three carbons and three oxygens.

If two of the four free valences of fragment **2b** are joined together, the remaining three carbons and three oxygens have to make a fragment with two valences, and this is a row of three carbonyls, generating structures **2c** and **2d**. These structures have to be dismissed, however, because they do not have two carbonyls within 2–3 bonds from both H6 and H8. If the four valences of **2b** are to be satisfied all by the three carbons and three oxygens, they have to make an oxalyl and a carbonyl group, generating structures **2e** and **2f**. Of these two structures, we choose **2f** as being by far the most likely one. The assignment of the two sides of the unsubstituted *para*-phenylene moiety followed from the strong nOe between protons 5.50 and 6.49.

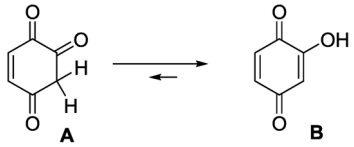
Ordinarily, triketones such as **2** of the general structure **A** (Scheme 3) are unstable relative to their hydroxyquinone tautomers (i.e., **B**)[8]. The extra strain that would be induced by enolization of **A** to form the second C–C double bond in the **B**-like tautomer is probably sufficient to favor the **A**-like tautomer present in paracyclophane **2**. Not unexpectedly for a compound like **A** containing a 1,3-diketone entity, the bridgehead methine proton of triketone **2**, H6, is reasonably acidic, and when dissolved in either acetone-d_6 or $CDCl_3$, it undergoes exchange with the solvent, and the signal at 2.23 ppm slowly disappears. In the ^{19}F spectra of partially deuterated compound **2**, one can observe the following deuterium isotope effects on ^{19}F chemical shifts: 800 ppb on the fluorine at –97.0, 90 ppb at

–109.7, 56 ppb at –114.8, 33 ppbat –120.6, –18 ppb at 103.3 and 5 ppb on the fluorine at –122.1.

The synthesis of **1** from AF4 comprises a rare example of direct, one-step oxidation of an aromatic system to a quinone. Generally, quinones are prepared by oxidation of phenols or similar derivatives[9]. As a result of this fortuitous reaction, AF4-quinone is much more readily accessible than the



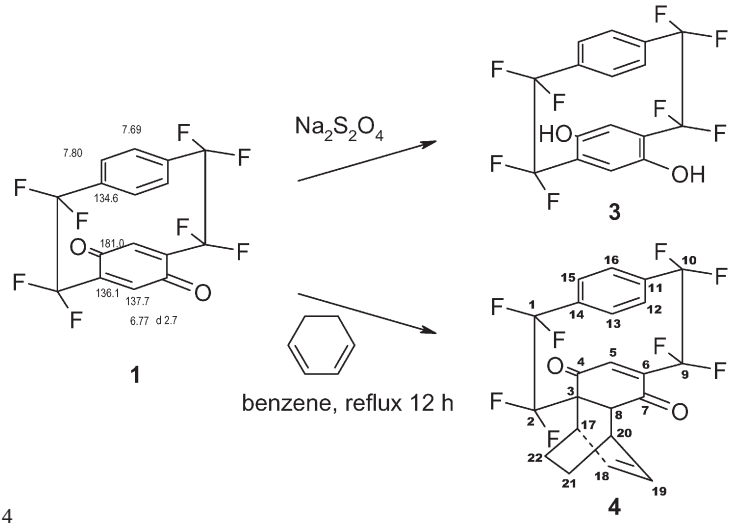
SCHEME 2
Fragments/structures considered for compound **2**



SCHEME 3

quinone of the analogous non-fluorine-containing [2.2]paracyclophane, which was first synthesized by Cram in a multistep process via the phenol precursor in an overall yield of <10% from the hydrocarbon[10]. Although improvements to the synthesis have been made[11,12], the hydrocarbon quinone remains relatively tedious to prepare.

AF4-quinone **1** is easily converted to the respective hydroquinone **3** in 88% yield by treatment with sodium dithionite, and, as with most quinones, it also acts as a reactive dienophile, as exemplified by its reaction with 1,3-cyclohexadiene to form adduct **4** in 78% yield (Scheme 4). NMR data for **4** are given in Table II. A strong nOe between 3.28 (H8) and 1.78 (H21) indicated that **4** is the expected *endo* Diels–Alder adduct.



SCHEME 4

Electrochemical characterization of AF4-quinone **1** was performed by cyclic voltammetry (CV) and differential pulse voltammetry (DPV) (Figs 1 and 2). The reduction potentials are listed in Table III along with potentials of related compounds. Similarly to 1,4-benzoquinone[13], the CV of AF4-quinone shows two chemically reversible reduction waves that are approximately 0.7 V apart although the reduction potentials of AF4-quinone are 0.25–0.3 V higher. The first reversible reduction wave at $E_{1/2}$ = –0.55 V (vs F_c/F_c^+) most likely corresponds to the formation of the radical anion of **1** by its one electron reduction. The second quasi-reversible reduction wave at $E_{1/2}$ = –1.27 V should be due to the formation of the hydroquinone dianion. The DPV experiments confirmed that both were one-electron processes. Because much of the literature references half wave potentials to

SCE, the measured reduction potentials for AF4-quinone (measured versus ferrocene) were each adjusted by +0.33 V for comparison purposes, these values now being E_1 = –0.22 V and E_2 = –0.94 V.

According to the literature[14], the analogous non-fluorinated quinone, [2.2]paracyclophane-4,7-dione (**5**) exhibits only one reduction wave as does

TABLE II
^{1}H, ^{13}C and ^{19}F chemical shifts of compound **4**, in benzene-d_6 at 25 °C

Position no.	$\delta^{13}C$ ppm	$\delta^{1}H$ or $\delta^{19}F$ ppm	Position no. of the protons which display cross-peaks in the gHMBC spectrum with the carbon in this position
1	115.2	–104.0, –115.5	13, 15
2	119.5	–104.6, –106.9	8
3	60.2	–	8, 17, 20, 22
4	192.2	–	[a]
5	141.4	–6.17	–
6	136.2	–	5
7	192.6	–	5, 8, 21[b]
8	52.6	3.28	19, 21
9	115.5	–114.0, –118.7	5
10	115.9	–118.8, –123.0	12, 16
11	134.5	–	13, 15
12	132.7	7.49	16
13	130.1	8.00	15
14	131.8	–	12, 16
15	129.6	7.73	13
16	129.5	7.78	12
17	40.0	3.36	18, 19, 21, 22
18	133.2	6.10	17, 20, 22
19	135.4	6.18	8, 17, 21
20	37.5	3.08	8, 18, 19, 21, 22
21	23.3	1.78, 1.38[c]	8, 17, 18, 19, 22
22	20.9	1.99, 1.16	17, 21

[a] Seen in the ^{13}C spectrum. [b] ^{4}J due to the planarity of the four bonds between H21 and C7. [c] The first value corresponds to the proton *anti* to the double bond in the norbornene moiety.

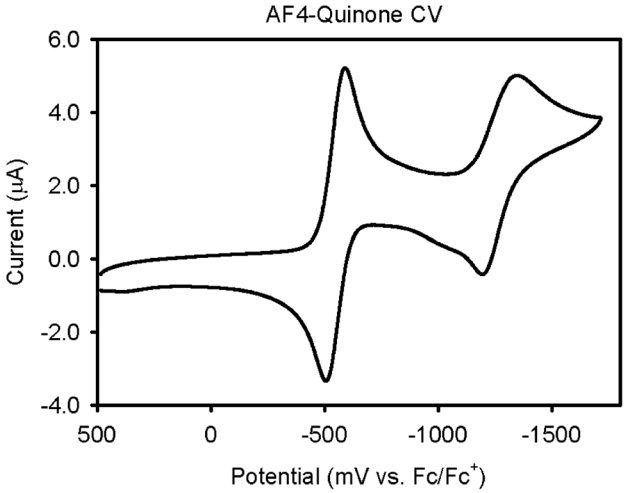


FIG. 1
Cyclic voltametry scan for AF4-quinone **1**

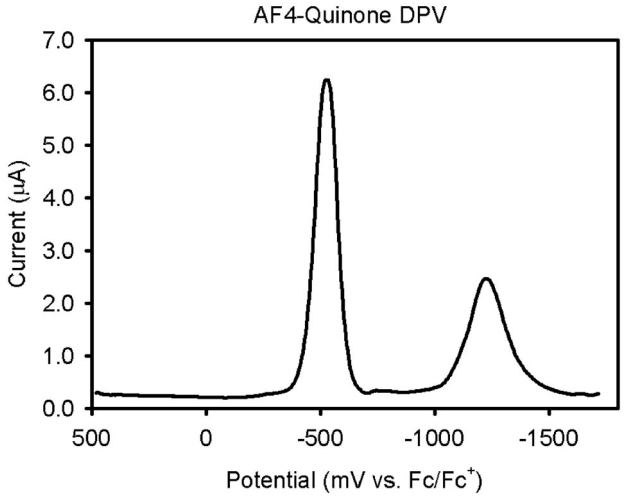


FIG. 2
Differential pulse voltammetry scan for AF4-quinone **1**

2,5-dimethyl-1,4-benzoquinone (Table III). In these cases, if these compounds follow a similar trend (i.e., $E_1 - E_2$ = ~0.7 V), then the second reduction likely occurs outside the potential window. Note that the reduction potential of [2.2]paracyclophane-4,7-quinone **5** is much lower than that of the similarly alkyl-substituted 2,5-dimethyl-1,4-benzoquinone. Most likely this difference derives from destabilization of the radical anion of **5** because of the relative non-planarity of its six-membered ring π-system.

The high reduction potential of **1** relative to **5** reflects the greater electron deficiency of the π-system of **1**. Its reduction potential is, however, lower than that of the analogous planar 2,5-bis(trifluoromethyl)-1,4-benzoquinone moiety, which indicates that, like the hydrocarbon system, the stability of the AF4-quinone radical anion must also suffer from its lack of planarity.

Comparing the reduction potential of **1** to those of other 1,4-benzoquinone systems, AF4-quinone appears to be most similar to the 2,5-dihalo-1,4-benzoquinones, as indicated by the values given for the 2,5-dichloro system in Table III.

UV spectra of **1** and **2** (in CH_2Cl_2) are given in Fig. 3. That of **1** exhibits λ_{max} values of 252 nm (ε = 10 800 cm^{-1} l mol^{-1}) and 292 nm (ε = 2 600 cm^{-1} l mol^{-1}), which is unexceptional for quinones. 1,4-Benzoquinone itself has a similar spectrum (in isooctane) with λ_{max} values of 240 nm (very strong) and 285 nm (weak)[16], as does the more closely related 2,5-bis(trifluoromethyl)-1,4-benzoquinone (λ_{max} 238 and 305 nm)[7]. There appears to

TABLE III
Reduction potential of AF4-quinone **1** and related compounds

Quinone	E_1, V[a]	E_2, V[a]	Ref.
AF4-quinone (**1**)	–0.22	–0.94	This work
[2.2]Paracyclophane-4,7-quinone (**5**)	–0.90	N/A	14
1,4-Benzoquinone	–0.55	–1.12	13
2-Methyl-1,4-benzoquinone	–0.56	–1.10	13
2,5-Dimethyl-1,4-benzoquinone	–0.67	–1.27	15
2-(Trifluoromethyl)-1,4-benzoquinone	–0.31	–0.98	15
2,5-Bis(trifluoromethyl)-1,4-benzoquinone	+0.05	–0.50	7
2,5-Dichloro-1,4-benzoquinone	–0.18	–0.81	15

[a] All potentials are given versus SCE.

be no evidence in the UV spectrum of **1** of the kind of charge-transfer band at a longer wavelength (at ~340 nm) that was claimed for [2.2]paracyclophane-4,7-quinone **5** [17]. Such charge transfer behavior was attributed to the close proximity of the second π-donor ring in the [2.2]paracyclophane system. The analogous π-system in **1** would not be a "donor" system.

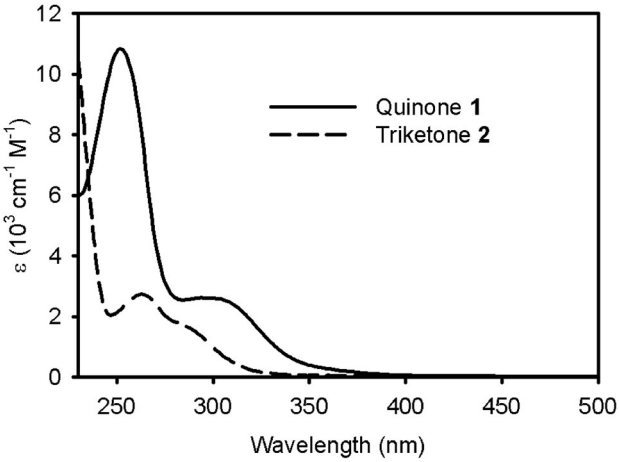


FIG. 3
UV spectra of AF4-quinone **1** and triketone **2**

CONCLUSIONS

Oxidation of AF4 with HIO_3 in trifluoroacetic acid has led to the direct synthesis of the respective *p*-quinone **1** along with a unique triketone **2**. The quinone behaves quite normally in its chemistry, being readily reduced to the respective hydroquinone and acting as a reactive dienophile in undergoing Diels–Alder reactions. Its reduction potential, as expected, is greater than that of its purely hydrocarbon analogue, with a reduction potential similar to 2,5-dichloro-1,4-benzoquinone.

EXPERIMENTAL

^{1}H-^{13}C correlation NMR spectra and the NOESY NMR spectra were recorded on a Varian Inova spectrometer equipped with a 5 mm indirect detection probe, operating at 500 MHz for ^{1}H and at 125 MHz for ^{13}C. Chemical shifts (δ) are reported in ppm relative to TMS, coupling constants (*J*) are given in Hz.

^{19}F and ^{19}F-^{19}F TOCSY and ^{1}H-^{19}F HOESY NMR spectra were recorded on a Varian Mercury spectrometer operating at 300 MHz for ^{1}H and 282 MHz for ^{19}F. The 5-mm conven-

tional probe had the high-band coil tuned simultaneously on ^{1}H and ^{19}F. ^{19}F chemical shifts are reported in ppm relative to $CFCl_3$.

The cyclic voltammetry (CV) experiments were performed on a Bioanalytical Systems CW50 electrochemical analyzer at a sweep rate of 100 mV s^{-1} using a platinum disc working electrode, a platinum wire auxiliary electrode, and a Ag|AgCl reference electrode. At the end of each scan, ferrocene was added as internal standard and potentials were referenced to the potential of ferrocene/ferrocenium redox couple. The differential pulse voltammetry experiments were performed with the same setup at a scan rate of 20 mV s^{-1}, pulse amplitude of 50 mV and pulse period of 200 ms. Sample and pulse width were 17 and 50 ms, respectively. Solutions of samples were prepared in dichloromethane. The supporting electrolyte was 0.10 M tetrabutylammonium hexafluorophosphate ($TBAPF_6$).

Oxidation of AF4

To a solution of sulfuric acid (1.23 g, 12 mmol) in trifluoroacetic acid (10 ml) were added AF4 (3.5 g, 10 mmol) and iodic acid (3.5 g, 20 mmol) at room temperature. The mixture was heated to reflux under stirring for 14 h. After cooling to room temperature, the mixture was poured into ice and water. The solid separated was collected and washed with water. The product was purified by flash chromatography (hexane/ethyl acetate 10:1 first, then 5:1) and followed by recrystallization from methylene chloride and hexane to give AF4-quinone **1** (2.15 g, 56%) and the triketone product **2** (0.80 g, 20%), both as yellow solids.

1,1,2,2,9,9,10,10-Octafluoro[2.2]paracyclophane-4,7-dione (**1**). M.p. 212–213 °C. ^{1}H NMR (300 MHz, acetone-d_6): 7.80 d, 2 H, ^{2}J = 8.1; 7.70 d, 2 H, ^{2}J = 8.1; 6.77 d, 2 H, ^{4}J = 2.7. ^{13}C NMR (75 MHz, acetone-d_6): 181.9 d, ^{3}J = 6.0; 137.7 dt, ^{4}J = 11.2, 3.8; 136.1 t, ^{2}J = 21.8; 134.6 t, ^{2}J = 26.2; 131.2 d, ^{4}J = 7.5; 129.9 t, ^{4}J = 7.7; 117.0 ddt, ^{1}J = 273.0, 266.2, ^{2}J = 27.0; 115.9 ddt, ^{1}J = 280.5, 263.2, ^{2}J = 30.4. ^{19}F NMR (282 MHz, acetone-d_6): –113.1 dd, ^{2}J = 240.5, ^{3}J = 6.2; –114.0 d, ^{2}J = 257.6; –117.0 dd, ^{2}J = 257.2, ^{3}J = 6.2; –119.2 d, ^{2}J = 238.6. For $C_{16}H_6F_8O_2$ (382.21) calculated: 50.28% C, 1.58% H; found: 50.58% C, 1.50% H.

Triketone **2**. M.p. 208–209 °C. ^{1}H NMR (500 MHz, benzene-d_6): 7.03 d, 1 H, ^{3}J = 8.4; 6.97 d, 1 H, ^{3}J = 8.9; 6.80 d, ^{3}J = 8.6; 6.49 d, 1 H, ^{3}J = 8.5; 5.50 s; 2.23 dt, 1 H, ^{3}J = 17.4, 3.4. ^{13}C NMR (125 MHz, benzene-d_6): 188.7, 185.8, 147.2, 133.9, 133.4, 131.4, 130.0, 127.4, 115.6, 114.9, 114.4, 52.9 (see Table I and the footnotes). ^{19}F NMR (282 MHz, benzene-d_6): –96.95 dddd , ^{2}J = 272.3, ^{3}J = 16.8, 15.3, 10.9; –103.3 dd, ^{2}J = 255.3, ^{3}J = 5.9; –109.8 d, ^{2}J = 271.5; –114.8 ddd, ^{2}J = 250.4, ^{3}J = 13.9, 5.0; –120.35 d, ^{2}J = 228.9; –120.6 dt, ^{2}J = 250.1, ^{3}J = 8.8; –121.5 d, ^{2}J = 228.9; –122.1 d, ^{2}J = 256.8. HMRS (M + H)$^{+}$: calculated for $C_{16}H_7F_8O_3$ 399.0267, found 399.0283. For $C_{16}H_6F_8O_3$ (398.21) calculated: 48.26% C, 1.52% H; found: 48.02% C, 1.59% H.

1,1,2,2,9,9,10,10-Octafluoro[2.2]paracyclophane-4,7-diol (**3**)

Under nitrogen, AF4-quinone **1** (0.240 g, 0.63 mmol) was dissolved in 1,2-dichloroethane (10 ml), the solution was heated to 40 °C and saturated aqueous sodium dithionite solution (1 ml) was added dropwise until the yellow color disappeared. After addition, the mixture was stirred at 40 °C for 30 min, then cooled to room temperature. The aqueous layer was extracted with 1,2-dichloroethane twice and the combined extracts were dried over anhydrous sodium sulfate. After removal of solvent, the residue was purified by flash chromatography (eluent: hexane/ethyl acetate 1:1) to give AF4-hydroquinone **3** as a pale white solid (0.214 g,

88%). M.p. 107–108 °C. ^{1}H NMR (300 MHz, acetone-d_6): 9.11 s, 2 H, broad; 7.78 d, 2 H, ^3J = 8.4; 7.19 d, 2 H, ^3J = 8.6; 6.35 s, 2 H. ^{13}C NMR (75 MHz, acetone-d_6): 150.3 d, ^3J = 6.8; 134.8 t, ^2J = 26.0; 129.8 dd, ^3J = 8.3, 5.8; 126.5 t, ^3J = 7.5; 121.7 t, ^2J = 26; 121.3 m; 119.3 ddt, ^2J = 263.6, 268.7, ^3J = 30; 119.2 ddt, ^2J = 272.5, 263.2, ^3J = 29.7. ^{19}F NMR (282 MHz, acetone-d_6): –112.4 d, ^2J = 239; –113.5 d, ^2J = 239; –114.7 d, ^2J = 236; –115.7 d, ^2J = 236. For $C_{16}H_8F_8O_2$ (384.22) calculated: 50.02% C, 2.10% H; found: 49.68% C, 2.09% H.

Diels–Alder Reaction of AF4-quinone with 1,3-Cyclohexadiene

Under nitrogen, a solution of AF4-quinone **1** (0.380 g, 10 mmol) and 1,3-cyclohexadiene (0.80 g, 10 mmol) in 10 ml of benzene was stirred under reflux for 12 h. After cooling to room temperature, the solvent was removed, and the residue was purified by flash chromatography to give the yellow solid product **4** (0.362 g, 78%). M.p. 236–237 °C. ^{1}H NMR (500 MHz, acetone-d_6): 8.00 d, ^3J = 8.3; 7.78 d, ^3J = 8.6; 7.73 d, ^3J = 8.5; 7.49 d, ^3J = 8.6; 6.18 t, ^3J = 7.8; 6.17 d, ^4J = 2.0; 6.10 t, ^3J = 7.3; 3.36 m; 3.28 m; 3.08 m; 1.99 t, ^3J = 11.4; 1.78 m; 1.38 tt, ^3J = 12.6, 3.4; 1.16 m. ^{13}C NMR (125 MHz, acetone-d_6): 192.5, 192.2, 141.4, 136.2, 135.4, 134.5, 133.2, 132.8, 131.8, 130.1, 129.7, 129.6, 116.0, 115.5, 115.3, 60.2, 52.6, 40.0, 37.5, 23.3, 20.9. ^{19}F NMR (282 MHz, acetone-d_6): –104.0 dd, ^2J = 252.2, ^3J = 15.7; –104.6 dd, ^2J = 256.1, ^3J = 8.4; –106.9 ddd, ^2J = 254.5, ^3J = 15.8, ^4J = 5.5; –114.0 dt, ^2J = 244.2, ^3J = 7.8; –115.5 d, ^3J = 252.5; –118.7 d, ^3J = 232.8; –118.7 d, ^3J = 242.2; –123.05 ddd, ^3J = 228.7, ^3J = 8.9, ^4J = 3.9. For $C_{22}H_{14}F_8O_2$ (462.33) calculated: 57.15% C, 3.05% H; found: 56.94% C, 3.36% H.

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