## SUPPORTING INFORMATION

# Dynamic HPLC Study of C<sub>70</sub> Chlorination Leads to a Surprisingly Selective Synthesis of C<sub>70</sub>Cl<sub>8</sub>

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Table 1.	Overview	of reported	C <sub>70</sub> chlorides. <sup>a</sup>
			- 10

No. [year] <sup>ref</sup>	proposed composition <sup>c</sup>	proposed purity [yield]	reagents and rxn. conditions	analytical techniques <sup>c</sup>	stability	solubility		
1 [1995] <sup>1</sup>	$C_{70}Cl_{10}^{\ b}$	n/r [qnt] <sup>c</sup>	ICl, C <sub>6</sub> H <sub>6</sub> , <sup>c</sup> reflx. <sup>d,e</sup>	IR, UV-vis, <sup>13</sup> C NMR	slow dcmp at RT	many org. slvnts		
2 [1996] <sup>2</sup>	C <sub>70</sub> Cl <sub>10</sub>	n/r. [n/r]	ICl, C <sub>6</sub> H <sub>6</sub> , heat <sup>f</sup>	n/r	n/r	$C_6H_6$		
3 [1998] <sup>3</sup>	$C_{70}Cl_{12}^{e}$	n/r. [n/r]	Cl <sub>2</sub> (liq.), RT	EA, TG, (Raman <sup>g</sup> )	$Cl_2$ loss at 200-300 °C	n/r		
4 [1999] <sup>4</sup>	$C_{70}Cl_{7-31}{}^{n}$	n/r [n/r]	Cl <sub>2</sub> , CCl <sub>4</sub> , UV	WG, MALDI-MS,	possible Cl <sub>2</sub> loss upon	CCl <sub>4</sub> , PhCH <sub>3</sub>		
				PIXE-NMP, EMP, HPLC	standing and/or analysis			
5 [2003] <sup>5</sup>	$C_{70}Cl_{10}^{\ \ h}$	n/r. [96%]	KICl <sub>4</sub> , ODCB, RT	EA, IR	n/r	n/r		
6 [2005] <sup>6</sup>	$C_{70}Cl_{28}{}^{i}$	n/r [n/r]	VCl <sub>4</sub> , SbCl <sub>5</sub> , PCl <sub>5</sub> , 140–200°C <sup>j</sup>	IR, <sup>k</sup> X-ray	stable on air po	oor sol. in org. slvnts		
7 [2005] <sup>7</sup>	$C_{70}Cl_{16}^{-1}$	n/r [n/r]	Br <sub>2</sub> , TiCl <sub>4</sub> , 60–80°C	IR, <sup>k</sup> X-ray	stable on air	n/r		
					Cl <sub>2</sub> loss at 240-360 °C	Cl <sub>2</sub> loss at 240-360 °C		
8 [2008] <sup>8</sup>	$C_{70}Cl_{8.4}Br_{1.6}{}^{m}$	n/r [n/r]	Br <sub>2</sub> , TiCl <sub>4</sub> , 80–90°C	IR, <sup>n</sup> X-ray	n/r	n/r		

<sup>a</sup> Abbreviations: mxt. = mixture; n/r = not reported; qnttv = quantitative; reflx. = reflux; ODCB = *o*-dichlorobenzene; EA = elemental analysis; UV = UVirradiation; ODCB = *o*-dichlorobenzene; PIXE-NMP = particle induced X-ray emission/nuclear microprobe analysis; EMP = electron microprobe analysis. <sup>b</sup> As determined by <sup>13</sup>C-NMR (based on symmetry requirements). <sup>c</sup> Quantitative yield of this synthesis was reported in ref. 4. <sup>d</sup> "Preparative details will be given in a full paper.<sup>n3</sup>; see item 2 [1996].<sup>4</sup> <sup>e</sup> In ref. 4 it was mentioned that this reaction took 20 minutes and was conducted in boiling C<sub>6</sub>H<sub>6</sub>; no other preparative details (like quantities of the reagents) were reported. <sup>f</sup> "... the mixture was heated until reaction was complete (ca. 20 min)." This description is ambiguous, but based on the ref. 4 it can be presumed that reaction was performed under reflux. <sup>g</sup> Raman spectroscopy was not informative due to strong fluorescence. <sup>h</sup> As determined by EA. <sup>i</sup> As determined by single-crystal X-ray diffraction study (the crystals were found to contain three isomers of C<sub>70</sub>Cl<sub>28</sub>). <sup>j</sup> C<sub>70</sub>Br<sub>10</sub> can also be used as a starting material, allowing to decrease the reaction time. <sup>k</sup> Experimental and calculated IR spectra of C<sub>70</sub>Cl<sub>28</sub> were reported. <sup>1</sup> As determined by single-crystal X-ray diffraction two isomers of C<sub>70</sub>Cl<sub>16</sub>). <sup>m</sup> As determined by single-crystal X-ray diffraction study. <sup>n</sup> IR spectroscopy results were mentioned, but the spectrum itself was not reported. Table S-1 presents the summary of the available literature data on the synthesis of  $C_{70}$  chlorides. The first work was published in 1995 by Sussex group, which reported the preparation and characterization of  $C_{70}Cl_{10}$  by ICl chlorination of  $C_{70}$  in benzene solution;<sup>1</sup> no information of the preparative procedure (such as concentrations of the reagents, reaction time and temperature, and yield and purity of the product) was given. A more detailed description of  $C_{70}Cl_{10}$  synthesis was given in a later paper (the yield and purity were not reported).<sup>2</sup>

In 1998 Ehrhardt et al. reported that  $C_{70}$  chlorination in liquid chlorine was achieved (reaction was performed in a sealed glass ampoule during a period of 30 days).<sup>3</sup> The elemental analysis of the product gave an average composition of  $C_{70}Cl_{12}$ ; the mass loss of this CF during thermogravimetry corresponded to much higher degree of chlorination of ca.  $C_{70}Cl_{21}$ .<sup>3</sup> The Raman spectroscopy of this product was not informative due to its strong fluorescence.<sup>3</sup>

In 1998 Heymann et al. reported the chlorination of  $C_{70}$  by chlorine in CCl<sub>4</sub> medium under UV irradiation.<sup>4</sup> The analysis of the  $C_{70}$ Cl<sub>n</sub> product by several analytical methods gave very different results on the degree of chlorination, which varied between n = 7 and n = 31. Chlorinated product was also analyzed by HPLC which demonstrated the absence of the starting material,  $C_{70}$ . However, authors did not report the number of the components observed in the HPLC trace (the corresponding HPLC trace was not shown in the paper<sup>4</sup>).

In 2003 Troshin et al. reported a synthesis of  $C_{70}Cl_{10}$  in 1,2-dichlorobenzene (ODCB) using KICl<sub>4</sub> chlorinating agent.<sup>5</sup> It was stated that independently of the amount of chlorinating agent  $C_{70}Cl_{10}$  was the only product formed (when less than stoichiometric amount of KICl<sub>4</sub> was used, mixtures of  $C_{70}Cl_{10}$  and unreacted  $C_{70}$  were obtained).<sup>5</sup>

In 2005, two higher  $C_{70}$  chlorides,  $C_{70}Cl_{28}$  and  $C_{70}Cl_{16}$  were reported; they were prepared in sealed ampoules using transition metal chlorides<sup>6</sup> and TiCl<sub>4</sub>/Br<sub>2</sub> mixture<sup>7</sup> correspondingly. These chlorides were structurally characterized by single-crystal X-ray diffraction study, which revealed that the crystals of both CFs contained several isomers of the corresponding composition (three isomers of  $C_{70}Cl_{28}^{-6}$  and two isomers of  $C_{70}Cl_{16}^{-7}$ ).

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**Figure S-1.** Dynamic HPLC plots of  $C_{70}$  chlorination (a) with 18 eq. ICl and (b) with 64 eq. ICl. The peaks marked with asterisks are due to the internal standard  $C_{60}F_{48}$ .



**Figure S-2.** Chlorination of 2.3 mM solution of  $C_{70}$  in PhCl by a large excess of ICl (1.13 M or 500 equivalents). The main peak is due to  $C_{70}Cl_{10}$ . Reaction time is given by the right side of the HPLC trace (100% toluene eluent was used at 5 mL/min flow rate).



**Figure S-3.** Top: reaction conditions, HPLC traces of the crude product and purified  $C_{70}Cl_8$  (insert) (80/20 v/v toluene/heptane eluent and 5 mL/min flow rate). Bottom: NI-APCI MS of purified  $C_{70}Cl_8$  (right), and experimental and calculated isotopic distribution of  $C_{70}Cl_7^-$  peak (left).



Figure S-4. MALDI mass spectra of 95+% pure C<sub>70</sub>Cl<sub>8</sub>.

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**Figure S-5.** UV-vis spectra of 95+% pure  $C_{70}Cl_8$  (1),  $C_s$ -(2) and  $C_2$ - $C_{70}(CF_3)_8$  (3).





**Figure S-7.** Synthesis and characterization of  $[C_{70}Cl_8]_n$  by thermogravimetry (5 °C/min heating rate, helium atmosphere) and negative- and positive-ion solvent-free MALDI mass spectrometry.



**Figure S-8.** HPLC traces (100% toluene, 5 mL/min flow rate) of  $C_{70}Cl_{10}$  samples prepared by our procedure (top) and by the literature procedure<sup>4</sup> (bottom). In the inserts, HPLC traces were acquired using 80/20 v/v toluene/heptane and 5 mL/min flow rate.



**Figure S-9.** Top figure: the literature IR spectrum of  $C_{70}Cl_{10}$  (pressed KBr pellet, see ref. 3). Bottom figure: the IR spectrum of 99%  $C_{70}Cl_{10}$  (pressed KBr pellet, this study).



**Figure S-10.** Synthesis, HPLC purification and analysis of  $C_{70}Cl_6$  (the small-size HPLC trace of the 95+%  $C_{70}Cl_6$  was acquired using 80/20 v/v toluene/heptane eluent and 5 mL/min flow rate). The peak markes with an asterisk may be due to lower chloride(s)  $C_{70}Cl_2$  and/or  $C_{70}Cl_4$ . NI-APCI- mass spectrum of the 95+% pure  $C_{70}Cl_6$  is shown below. The bottom part of the figure shows the experimental and calculated isotopic distribution of  $C_{70}Cl_6^-$  peak.



**Figure S-11.** UV-vis spectra of 95+%  $C_{70}Cl_6$  and  $C_2$ - $C_{70}(CF_3)_6$ . The likely  $C_2$ -addition pattern of  $C_{70}Cl_6$  is highlighted by the red square.



**Figure S-12.** Dynamic HPLC study of the stability  $C_{70}Cl_{10}$  (top),  $C_{70}Cl_8$  (middle), and  $C_{70}Cl_6$  (bottom) in toluene solution. Four identical samples I-IV were prepared for each CF. Samples I were stored under nitrogen in the dark; samples II were stored under air in the dark; samples III were stored under air under light. under different conditions. The peak on the right side of the main peak of  $C_{70}Cl_{10}$  is due to  $C_{70}Cl_8$  impurity.

### **Experimental Details**

Reagents and Solvents: Benzene (Sigma-Aldrich, Na), toluene (Fischer Scientific, Na), chlorobenzene (Sigma-Aldrich, CaH<sub>2</sub>), fluorobenzene (Sigma-Aldrich, CaH<sub>2</sub>), and 1,2dichlorobenzene (Sigma-Aldrich, CaH<sub>2</sub>) were ACS Reagent Grade (vendor indicated in parenthesis) and were distilled from the indicated drying agent under purified N2 atmosphere prior to use. TiCl<sub>4</sub> (Sigma-Aldrich) was stirred with copper powder for several days, then distilled under vacuum. HPLC Grade toluene, heptanes (Fisher Scientific), and CH<sub>2</sub>Cl<sub>2</sub> (Fisher Scientific) were used as received. C<sub>60</sub> (99.9%, Term-USA), iodine monochloride (Sigma Aldrich, 99.998% metals trans-2-[3-{4-tert-butylphenyl}-2-methyl-2trace basis), propenylidene]malononitrile (Fluka), chromium(iii) acetyl acetonate (Sigma Aldrich), and KBr (Sigma Aldrich, 99+ % FTIR grade) were used as received. All syntheses were carried out under a purified N<sub>2</sub> atmosphere by using standard Schlenk techniques with vigorous stirring by a magnetic stirrer.

**Instruments.** HPLC analysis and separation was done using Shimadzu liquid chromatography instrument (CBM-20A control module, SPD-20A UV-detector set to 300 nm detection wavelength, LC-6AD pump, manual injector valve) equipped with 10-mm I.D.  $\times$  250 mm Cosmosil Buckyprep column, Nacalai Tesque, Inc.). Electronic spectra of dichloromethane and/or toluene solutions of chlorofullerenes were recorded using a Varian Cary 500 spectrophotometer. MALDI mass spectra were recorded on a Kompact MALDI IV (Kratos Analytical, Manchester, UK) time-of-flight mass-spectrometer in the linear mode. A 337 nm N<sub>2</sub> laser was used for target activation. Each mass spectrum was the average of 50–100 laser shots. CF samples and the trans-2-[3-{4-tert-butylphenyl}-2-methyl-2-propenyl-idene]malononitrile matrix material (DCTB) were dissolved separately in toluene and were mixed in a 1:10 mol/mol sample/DCTB ratio assuming the sample contained only C<sub>70</sub>Cl<sub>10</sub>. A drop of each sample/DCTB solution was deposited on a stainless steel slide by using a capillary and dried under a strong stream of cool air from an airsprayer/brush in order to achieve a uniform sample surface. MALDI mass spectra of the insoluble  $[C_{70}Cl_8]_n$  dimer were obtained by solvent-free MALDI as described in S.G. Kotsiris et al. *Eur. J. Mass Spectrom.* **2006**, *12*, 397. The molar analyte-tomatrix ratio was approximately 1:25. APCI mass-spectra were recorded on 2000 Finnigan LCQ-DUO mass-spectrometer (CH<sub>3</sub>CN carrier solvent, 0.3 mL/min flow, CF sample injected as solution in toluene). Thermogravimetry was performed using a TA Instruments TGA-2950 (platinum sample pans, *ca.* 5 mg sample size, 25–500 °C temperature range).

 $C_{60}F_{48}$ . 98+% pure  $C_{60}F_{48}$  was prepared following the procedure described in A. A. Popov et al. *Phys. Chem. A* **2006**, *110*, 8645. It was used without further purification.

**Iodine Monochloride Handling and Transfer.** In a typical experiment, the storage container with solid ICl (a storage tube equipped with a Teflon valve and a side arm) was warmed up, the resulted liquid then measured and transferred using an air-tight syringe (50, 250, and 500  $\mu$ L syringes were used) equipped with a Teflon straw of sufficient volume to accommodate all of ICl (to avoid ICl contact with the stainless steel needle and a possible metallic contamination) under protective flow of purified N<sub>2</sub>. DANGER! ICl is very volatile, extremely corrosive to metal and rubber, and moisture-sensitive.

**CF Handling.** All operations involving solutions of CFs were performed either in the dark (vessels containing CF solutions were wrapped with aluminum foil) or with minimal exposure to light (experimental operations were performed as quickly as possible under minimal illumination) unless reported otherwise. We recommend that prior to long-term storage the traces of aromatic solvents (toluene etc.) should be removed by dissolving a CF sample in HPLC grade  $CH_2Cl_2$  and evaporating the resulting solution under vacuum (see Appendix A.I.1).

**Preparation of**  $C_{70}$ **Cl**<sub>10</sub>. C<sub>70</sub> solution (83.0 mg, 0.099 mmol) in dry, deoxygenated CB (45 mL) was mixed with ICl (1.25 mL, 4.00 g, 24.6 mmol, 250 eq.) under vigorous stirring at ca. 20 °C in the glass reactor of local design (see Figure II.19 and I. V. Kuvychko et al. *J. Am. Chem. Soc.* **2010,** 132, 6443). The evaporation chamber of the apparatus was immersed in a room-temperature water bath. After 7 min the reactor was evacuated (the trap was pre-cooled with

liquid nitrogen) and the volatiles were quickly removed. Longer reaction times (up to tens of minutes) are unlikely to cause a detectable decrease of the product purity. The product was washed off the walls of the evaporation chamber with a small volume of HPLC grade gave  $CH_2Cl_2$  and transferred into a storage vessel, and then  $CH_2Cl_2$  was removed under vacuum. This procedure 117.9 mg (0.0986 mmol, 99% yield) of 98%  $C_{70}Cl_{10}$  (according to the integration of the HPLC trace collected with 100% toluene eluent at 5 mL/min flow rate).

**Preparation of C**<sub>70</sub>**Cl**<sub>10</sub> (Sussex group procedure: Birkett, et al. J. Chem. Soc., Chem. Commun. 1995, 683; Birkett, et al. Chem. Commun. 1996, 1231).

). ICl (0.125 mL, 400 mg, 2.46 mmol, 180 eq.) was added to a vigorously stirred solution of C<sub>70</sub> (11.6 mg, 0.0138 mmol) in C<sub>6</sub>H<sub>6</sub> (36 mL). Then reaction mixture was heated to ca. 70 °C using preheated oil bath for 20 minutes. After heating the reaction mixture was set to cool down for 10 minutes. Then the reaction mixture was split into two parts (A and B). The part A was worked up using the original aqueous method of Sussex group. The part B of the reaction mixture was evaporated down using our evaporation apparatus. The products obtained by these two procedures were dissolved completely in toluene (no insoluble products were observed); the resulting solutions were analyzed by HPLC (100% toluene eluent at 5 mL/min flow rate). The HPLC analysis of products A and B showed only minor differences in the distribution of products. Both products contained C<sub>70</sub>Cl<sub>8</sub> as the major constituent (ca. 70% according to HPLC trace integration; 80/20 toluene/heptane eluent was used) and ca. 20% of C<sub>70</sub>Cl<sub>10</sub> (the other ca. 10% being composed of C<sub>70</sub>).

**Preparation of C**<sub>70</sub>**Cl**<sub>8</sub>. ICl (73  $\mu$ L, 233 mg, 1.44 mmol, 50 eq.) was added to a vigorously stirring solution of C<sub>70</sub> (24.4 mg, 0.0290 mmol) in CB (15 mL) in evaporation apparatus. The evaporation chamber of the apparatus was immersed in a room-temperature water bath. After 90 sec the vacuum was applied and the volatiles were removed (the evaporation took ca. 2 min). The brown residue was kept under dynamic vacuum for additional 30 minutes. The crude product was dissolved in toluene (the absence of the insoluble residue demonstrates that no

significant amount of  $[C_{70}Cl_8]_n$  was formed) and  $C_{70}Cl_8$  was separated from  $C_{70}$  impurity by HPLC (100% toluene eluent, 5 mL/min). The yield of  $C_{70}Cl_8$  is ca. 80% (based on HPLC trace integration of the crude product). No Carbon-13 NMR spectroscopic study was performed due to a relatively low solubility of  $C_{70}Cl_8$  and its tendency to dimerize and precipitate in concentrated solutions.

**Preparation of**  $[C_{70}Cl_8]_n$ . ICl (200 µL, 640 mg, 3.94 mmol, 50 eq.) was added to a vigorously stirring solution of C<sub>70</sub> (71.1 mg, 0.0846 mmol) in CB (44 mL) in evaporation apparatus. The evaporation chamber of the apparatus was immersed in a room-temperature water bath. After 25 min the vacuum was applied and the volatiles were removed (the evaporation took ca. 8 min). The brown residue was washed off the walls of the reactor with ca. 3 mL of HPLC grade toluene. The resulting slurry was centrifuged and the brown supernatant was decanted. The insoluble residue was washed again with toluene and two more times with HPLC grade dichloromethane Centrifugation and decantation were used instead of filtration to avoid losses. The resulting lightbrown solid was dried under vacuum to remove traces of solvents, giving 71.1 mg of  $[C_{70}Cl_8]_n$  (0.0316 mmol, 75% yield).

**Preparation of C**<sub>70</sub>**Cl**<sub>6</sub>. ICl (12  $\mu$ L, 36 mg, 0.22 mmol, 14 eq.) was added to a vigorously stirring solution of C<sub>70</sub> (14.0 mg, 0.0167 mmol) in CB (11 mL) in evaporation apparatus. The evaporation chamber of the apparatus was immersed in a room-temperature water bath. After 1 hour and 30 minutes the vacuum was applied and the volatiles were removed (the evaporation took ca. 5 min). The brown residue was kept under dynamic vacuum for additional 30 minutes. The crude product was dissolved in 80/20 v/v toluene/heptane mixture and C<sub>70</sub>Cl<sub>6</sub> was separated by HPLC (80/20 v/v toluene/heptane eluent, 5 mL/min). The yield of C<sub>70</sub>Cl<sub>8</sub> is ca. 27% (based on HPLC trace integration of the crude product). No Carbon-13 NMR spectroscopic study was performed due to a relative low solubility of C<sub>70</sub>Cl<sub>6</sub>.

**Dynamic HPLC study of C**<sub>70</sub> **chlorination.** A stock solution of C<sub>70</sub> and C<sub>60</sub>F<sub>48</sub> in PhCl (solution 1) was prepared by adding C<sub>70</sub> (25.0 mg) and C<sub>60</sub>F<sub>48</sub> (27.0 mg) to 10 mL of dry, deoxygenated

PhCl. After ca. 24 hours of stirring the solution was filtered. A stock solution of ICl in  $CH_2Cl_2$  was prepared by dissolving ICl (312 µL, 1.00 g, or 0.62 mM) 10.0 mL of dry, deoxygenated  $CH_2Cl_2$ . The experiments were performed in the small-scale greaseless reactors of local design.

**Experiment a.** 2.0 mL of stock solution 1 were mixed with stock solution 2 (136  $\mu$ L). Small samples of the reaction mixture were taken at regular time intervals, flash-evaporated under vacuum, dissolved in toluene and analyzed by HPLC (100% toluene eluent, 5 mL/min flow rate). Initial concentrations of the reagents: [C<sub>70</sub>] = 2.3 mM; [ICl] = 42 mM (18 eq.).

**Experiment b.** 2.0 mL of stock solution 1 were mixed with ICl (15  $\mu$ L). Small samples of the reaction mixture were taken at regular time intervals, flash-evaporated under vacuum, dissolved in toluene and analyzed by HPLC (100% toluene eluent, 5 mL/min flow rate). Initial concentrations of the reagents: [C<sub>70</sub>] = 2.3 mM; [ICl] = 147 mM (64 eq.).

**Experiment c.** 2.0 mL of stock solution 1 were mixed with ICl (115  $\mu$ L). Small samples of the reaction mixture were taken at regular time intervals, flash-evaporated under vacuum, dissolved in toluene and analyzed by HPLC (100% toluene eluent, 5 mL/min flow rate). Initial concentrations of the reagents: [C<sub>70</sub>] = 2.3 mM; [ICl] = 1.13 M (500 eq.).

**Photodegradation Experiments:**  $C_{70}Cl_{10}$ /toluene. A sample of 2.7 mg of  $C_{70}Cl_{10}$  (with ca. 15%  $C_{70}Cl_8$  contamination) was dissolved in 30 mL of dried, deoxygenated toluene under dry  $N_2$  atmosphere. This solution was split into four parts of approximately same volume. Parts I and II were transferred into Schlenk flasks (under  $N_2$ ), when parts III and IV were transferred into 25 mL volumetric flasks with small-diameter necks and ground-glass stoppers (all reservoirs were made of colorless Pyrex glass). Solutions III and IV were aerated for 10 sec by bubbling air through them. The flasks containing solutions I and III were wrapped in aluminum foil to shield them from light, while the flasks with solutions II and IV were left in the fume hood exposed to a continuous irradiation with ambient fluorescent light. All solutions were stored in the same place to ensure their equal temperature. All four flasks were tightly capped (using silicon grease) to avoid solvent loss and air contamination. Samples of the solutions I, II, III, and IV were taken

(under protective flow of N<sub>2</sub> in case of samples **I** and **II**) regularly and analyzed immediately by HPLC (100% toluene eluent, flow 5 mL/min, 300 nm detection wavelength,  $Ø10 \text{ mm} \times 250 \text{ mm}$ semipreparative Cosmosil BuckyPrep column, 500 µL injection volume).

**Photodegradation Experiments:**  $C_{70}Cl_8$ /toluene. A sample of ca. 2 mg of HPLC purified 95+%  $C_{70}Cl_8$  was dissolved in 20 mL of dried, deoxygenated toluene under dry N<sub>2</sub> atmosphere. This solution was treated analogously to the solution of  $C_{70}Cl_{10}$ /toluene in the corresponding photodegradation experiment.

**Photodegradation Experiments:**  $C_{70}Cl_6$ /toluene. A sample of ca. 1 mg of HPLC purified 95+%  $C_{70}Cl_6$  was dissolved in 14 mL of dried, deoxygenated toluene under dry N<sub>2</sub> atmosphere. This solution was treated analogously to the solution of  $C_{70}Cl_{10}$ /toluene in the corresponding photodegradation experiment.

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