Effect of Halogen Substitution on Energies and Dynamics of Reversible Photomechanical Crystals Based on 9-Anthracenecarboxylic Acid

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Figure S1: Complete fluorescence recovery for fluorinated anthracenes

Figure S2: Monoclinic and triclinic 9AC FRAP

S3: Synthesis of 4,5diF-9AC, 2Cl-9AC, 4Cl-9AC, 4,5diCl-9AC

S4: Crystallographic information for 4,5diF-9AC

S5: Crystallographic information for 4,5diCl-9AC



Figure S1: Fluorescence recovery data on long timescales for compounds: a) 9AC, b) 2F-9AC, c) 10F-9AC, and d) 2,6-diF-9AC. Red points are for \sim 50% photoconversion, while black points are for \sim 10% photoconversion, all with intermittent probe. Continuous lines show data for continuous probe exposure, which tends to slow down the recovery for large photoconversions.



Figure S2: The fluorescence recoveries of monoclinic and triclinic 9AC polymorphs are effectively identical for both a) 30% photodimerization and b) 60% photodimerization. Note the different time axes.

S3: Synthesis of 4,5diF-9AC, 2Cl-9AC, 4Cl-9AC, 4,5diCl-9AC

<u>Materials</u>: 9-Anthracene carboxylic acid (9AC, 99%), 1-Chloroanthracene (95%), Cesium Fluoride (99%, anhydrous), Zinc (99.9%, powder), Lithium (99%, ribbon), and 1-Bromobutane (99%), and n-Butyllithium (2.5 M hexanes) were purchased from Merck Sigma-Aldrich and used without further purification. 1,8-Dichloroanthraquinone (95% GC) was purchased from TCI-America and used without further purification. 2-Chloroanthracene was purchased from Santa Cruz Biotechnology and used without further purification. Dry diethyl ether (Et₂O) was prepared by distillation over LiAlH₄ and stored over activated molecular sieves (4 Å). All other organic solvents were distilled over CaH₂ and stored over activated molecular sieves (4 Å). Glassware were kept dry in an oven at 60 °C prior to use. <u>Equipment</u>: Melting point were determined using Stuart Melting Point SMP30 inside sealed capillary tubes

Synthesis of 4,5-Difluoroanthracene-9-carboxylic acid (4,5diF-9AC): Synthetic Scheme-1 was followed



Scheme 1: The synthesis of 4,5diF-9AC

Synthesis of 1,8-Difluoro-anthraquinone: The use of dry CsF is crucial for the chemical yield and purity of 1,8-Difluoro-anthraquinone. CsF (4 g, 26 mmole) was placed in a Schlenk tube (25 ml) and heated to 200 °C for two hours under vacuum (5 mbar). The anhydrous CsF was cooled to room temperature under a blanket of Argon gas. 1,8-Dichloroanthraquinone (1.1 g, 4 mmoles) was mixed thoroughly with the anhydrous CsF using a mortar and pestle while gently purging the mixture under a stream of Argon gas. The dry mixture was transferred into a small 50 ml tall form beaker, sealed with aluminum foil and placed over a hot plate set at 280 °C for 15 hours. 1,8-Difluoro-anthraquinone sublimes and deposits in the form of yellow needle crystals on the walls of the beaker. Obtained yellow crystalline needles (0.6g, 62% yield) with a mp of 227-229 °C (literature mp: 228 °C)¹. ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 8.03

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 $(2H, dd, J = 7.8 Hz, 0.9Hz), 7.79 (2H, ddd, J_{H-F} = 4.6 Hz, J = 7.8 Hz, 8.2 Hz), 7.54 (2H, ddd, J_{H-F} = 11 Hz, 8.2 Hz, 0.9 Hz).$



¹H NMR of 1,8-Difluoroanthraquinone in 10% DMSO-d₆ in CCl₄

Synthesis of 1,8-Difluoroanthracene: Into a 100 mL round-bottom flask was suspended 1,8-Difluoroanthraquinone (0.4 g, 1.6 mmoles) in 27% NH₄OH solution (10 mL), water (10 mL). Zinc powder (1.1 g, 17 mmoles), prewashed with dilute hydrochloric acid and dried, was added to the mixture along with catalytic amount of CuSO₄ (0.05 g) and stirred vigorously at 80 °C. The flask was fitted with an aircooled Snyder column that allows excess ammonia gas to escape while stopping air from flowing back inside the flask. The reaction changes from deep red to a gray after 24 to 36 h or when all the 1,8-Difluoro-anthraquinone is reduced to the corresponding 1,8-Difluoroanthracene. The progress of the reaction was monitored by TLC using Ethyl acetate: Hexanes (1:4) as mobile phase. The reaction mixture was cooled to room temperature and concentrated HCl solution was slowly added till all the excess Zn and ZnO dissolve and the solution becomes acidic. The product was extracted with ethyl acetate (40 ml) followed by washing the organic phase with brine and drying using anhydrous MgSO₄. The organic phase was decanted and the solvent removed under reduced pressure to obtain light yellow crude solid. Recrystallization from boiling ethanol/ water offers pale-yellow crystals (0.18 g, 52% yield) mp of 140-142 °C (literature mp: 142°C²). ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 8.83 (1H, s), 8.49 (1H, s), 7.78 (2H, d, J_{H-F} = 8.5 Hz), 7.40 (2H, dd, J_{H-F} = 7.8 Hz, 2.3Hz), 7.1 (2H, dd, J_{H-F} = 10.5 Hz, 7.3 Hz)



 $^1\mathrm{H}$ NMR of 1,8-Difluoroanthracene in 10% DMSO-d_6 in CCl_4

Synthesis of 10-Bromo-1,8-Difluoroanthracene: Bromine (0.18 g, 1.1 mmoles) was dissolved in CCl₄ (5 ml) and added slowly to a solution of 1,8-Difluoroanthracene (0.12 g, 0.56 mmoles) in CCl₄ (5 ml). The mixture was stirred at room temperature for 2 hours followed by overnight stirring at 60 °C. The reaction mixture was passed through a silica gel plug (5 g, flash chromatography grade) and eluted with hexanes. The organic solvent was removed under reduced pressure to obtain yellow crystals of 10-Bromo-1,8-Difluoroanthracene (0.15 g, 91% yield) with a mp of 134-136 °C (recrystallized from Ethanol/ water). ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 8.92 (1H, s), 8.24 (2H, d, J_{H-F} = 9.2 Hz), 7.56 (2H, ddd, J_{H-F} = 5.6 Hz, 9.2 Hz, 7.6 Hz), 7.22 (2H, dd, J_{H-F} = 10 Hz, 7.6 Hz).



¹H NMR of 10-Bromo-1,8-Difluoroanthracene in DMSO-d₆

Synthesis of 4,5-Difluoroanthracene-9-Carboxylic acid (4,5diF-9AC): In a 50 mL oven dried Schlenk flask, 10-Bromo-1,8-Difluoroanthracene (0.2 g, 0.73 mmoles) was dissolved in dry diethyl ether (20 ml) and cooled to < -50 °C using previously cooled Lab Armor dry bath beads. n-Butyllithium (2.5 molar in hexane, 0.32 ml, 1.1 equivalent) was added to the solution and allowed to react for 1 hour while maintaining the temperature < -50 °C. Dry CO₂ gas was bubbled for 20 minutes while the reaction was allowed to slowly warm to room temperature. The reaction mixture was quenched by adding it to crushed ice (50 ml) followed by extracting the aqueous phase with ethyl acetate (50 ml) followed by hexanes (2 x 50 ml). The carboxylic acid was precipitated out of the aqueous phase by adding concentrated HCl (3 ml) to offer a yellow precipitate of crude 4,5diF-9AC that was suction filtered and recrystallized from boiling ethanol (10 ml) and water (10 ml). Obtained a yellow crystalline powder (0.22 g, 85% yield) with a mp of 274-275 °C . ¹H NMR (400 MHz, Acetone-d₆) δ (ppm): 8.97 (H, s), 8.01 (2H, d, J_{H-F} = 8.9 Hz), 7.64 (2H, ddd, J_{H-F} = 5.8 Hz, 8.9 Hz, 7.3 Hz), 7.37 (2H, dd, J_{H-F} = 10.8 Hz, 7.3 Hz). ¹³C NMR (100 MHz, Acetone-d₆) δ (ppm): 168.77 (C=O), 159.92-157.40 (C-F, J = 252 Hz), 129.88, 129.30, 127.55-127.47 (C-C-C-F, J = 8 Hz), 122.19, 121.61-121.57 (J = 4 Hz), 114.35, 108.96-108.77 (C-C-F, J = 20 Hz).



¹H NMR of 4,5-Difluoroanthracene-9-Carboxylic acid in Acetone-d₆



 $^{13}\mathrm{C}$ NMR of 4,5-Difluoroanthracene-9-Carboxylic acid in Acetone-d_6

Synthesis of 2-Chloroanthracene-9-carboxylic acid (2Cl-9AC): Synthetic Scheme-2 was followed



Scheme 2: The synthesis of 2Cl-9AC

Synthesis of 9-Bromo-2-chloro-anthracene: Bromine (0.15 g, 0.56 mmoles, 1 eq.) was dissolved in CCl₄ (5 ml) and added slowly over a period of 10 min to a solution of 2-Chloroanthracene (0.2 g, 0.56 mmoles, 1 eq.) in CCl₄ (40 ml). The mixture was stirred at room temperature for 2 hours followed by overnight stirring at 60 °C. The reaction mixture was passed through a silica gel plug (5 g, flash chromatography grade) and eluted with hexanes. The organic solvent was removed under reduced pressure to obtain relatively pure yellow crystals of 9-Bromo-2-chloro-anthracene (0.26 g, 94% yield). A small quantity was recrystallized from acetic acid to give yellow crystals with mp of 138.5-141 °C (literature mp: 139-140 °C)³. ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 8.50 (1H, s), 8.43 (2H, d, J = 8.9 Hz), 8.02 (2H, dd, J = 3.4 Hz, 8.9 Hz), 7.61 (1H, ddd, J = 0.9 Hz, 1.4 Hz, 6.8 Hz), 7.52 (1H, ddd, J = 1.4 Hz, 1,1 Hz, 8.2 Hz), 7.41 (1H, dd, J = 1.8 Hz, 9.1 Hz)



¹H NMR of 9-Bromo-2-chloro-anthracene in DMSO-d₆

Synthesis of 2-Chloro-anthracene-9-carboxylic acid (2Cl-9AC): In a 50 mL oven dried Schlenk flask, 9-Bromo-2-chloro-anthracene (0.23 g, 0.78 mmoles) was dissolved in dry diethyl ether (20 ml) and cooled to -50 °C using previously cooled Lab Armor dry bath beads. n-Butyllithium (2.5 molar in hexane, 0.31 ml, 1.0 equivalent) was added to the solution and allowed to react for 1 hour while maintaining the temperature < -50 °C. Dry CO₂ gas was bubbled for 20 minutes while the reaction was allowed to slowly warm to room temperature. The reaction mixture was quenched by adding it to crushed ice (50 ml) followed by extracting the aqueous phase with ethyl acetate (50 ml) followed by hexanes (2 x 50 ml). The carboxylic acid was precipitated out of the aqueous phase by adding concentrated HCl (3 ml) to offer a yellow precipitate of crude 2Cl-9AC that was suction filtered and recrystallized from boiling ethanol/ water. Obtained a yellow crystalline powder (0.13 g, 65% yield) with a mp of 226-228 °C . ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 8.77 (1H, s), 8.20 (1H, d, J = 9.6 Hz), 8.14 (1 H, d, J = 8.9 Hz), 8.03 (1H dd, J = 1.1 Hz, 0.9 Hz), 7.61 (3H, m). ¹³C NMR (100 MHz, DMSO-d₆) δ (ppm): 170.12 (C=O), 132.44, 131.72, 131.29, 129.70, 129.32, 129.18, 128.45, 128.26, 127.69, 126.97, 126.71, 125.45, 123.49



 $^1\mathrm{H}$ NMR 2-Chloro-anthracene-9-carboxylic acid in DMSO-d_6



 $^{13}\mathrm{C}$ NMR 2-Chloro-anthracene-9-carboxylic acid in DMSO-d_6

Synthesis of 4-Chloroanthracene-9-carboxylic acid (4Cl-9AC): Synthetic Scheme-3 was followed



Scheme 3: The synthesis of 4Cl-9AC

Synthesis of 10-Bromo-1-chloro-anthracene: Synthesis of this compound was done using a procedure similar to the one used for the synthesis of Synthesis of 9-Bromo-2-chloro-anthracene. Obtained yellow crystals (92% yield) with a mp = 132-134 °C. ¹H NMR (DMSO-d₆) δ (ppm): 8.95 (1H, s), 8.39 (2H, dd, J = 8.9 Hz, 0.7 Hz), 8.29 (1H, dd, J = 0.7 Hz, 9.1 Hz), 7.77 (2H), 7.63 (2H, m)



¹H NMR 10-Bromo-1-chloro-anthracene in DMSO-d6

Synthesis of 4-Chloro-anthracene-9-carboxylic acid (4Cl-9AC): Synthesis of this compound was done using a procedure similar to the one used for the synthesis of Synthesis of 2-Chloro-anthracene-9-carboxylic acid. Obtained yellow crystals (60% yield) with a mp = 256 °C with decomposition (Literature

mp = 258 °C⁴). ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 8.97 (1H, s), 8.30 (1H, d, J = 8.2 Hz), 8.09 (2H, dd, J = 1.4 Hz, 8.9 Hz), 7.76 (1H, dd, J = 0.7 Hz, 7.1 Hz), 7.63 (2H, m), 7.55 (1H, dd, J = 8.9 Hz, 7.3 Hz). ¹³C NMR (100 MHz, DMSO-d₆) δ (ppm): 170.33 (C=O), 131.73, 131.61, 129.66, 128.76, 128.57, 128.08, 128.04, 127.75, 127.25, 127.16, 126.57, 126.22, 125.31, 125.26



¹H NMR 4-Chloro-anthracene-9-carboxylic acid in DMSO-d6



¹³C NMR 4-Chloro-anthracene-9-carboxylic acid in DMSO-d6

Synthesis of 4,5-dichloroanthracene-9-carboxylic acid (4,5diCl-9AC): Synthetic Scheme 4 was followed



Scheme 4: The synthesis of 4,5diCl-9AC

Synthesis of 1,8-Dichloroanthracene: Synthesis of this compound was done following the literature procedure.⁵ Obtained yellow needles (51% yield) with a mp = 157-158 °C (Lit. 156.5-158 °C⁵). ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 9.17 (1H, s), 8.54 (1H, s), 7.97 (2H, d, J = 8.5 Hz), 7.62 (2H, d, J = 7.3 Hz), 7.43 (2H, dd, J = 7.1 Hz, 8.5 Hz)



¹H NMR 1,8-Dichloroanthracene in DMSO-d₆

Synthesis of 10-Bromo-1,8-dichloroanthracene: Synthesis of this compound was done following the literature procedure.⁶ Obtained yellow crystals (82% yield) with a mp = 199-201 °C. ¹H NMR (DMSO- d_6) δ (ppm): 9.32 (1H, s), 8.46 (2H, dd, J = 8.9 Hz, 0.9 Hz), 7.71 (2H, d, J = 7.1 Hz), 7.58 (2H, dd, J = 7.3 Hz, 8.9 Hz)



¹H NMR 10-Bromo-1,8-dichloroanthracene in DMSO-d₆

Synthesis of 4,5-dichloroanthracene-9-carboxylic acid (4,5diCl-9AC): Synthesis of this compound was done using a procedure similar to the one used for the synthesis of Synthesis of 4-Chloro-anthracene-9-carboxylic acid. Obtained yellow crystals (40% yield) with a mp > 270 °C. ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 9.19 (1H, s), 8.02 (2H, ddd, *J* = 0.9 Hz, 1.8 Hz, 8.9 Hz), 7.87 (2H, d, *J* = 6.6 Hz), 7.65 (2H, dd, *J* = 7.2 Hz, 8.7 Hz). ¹³C NMR (100 MHz, DMSO-d₆) δ (ppm): 169.88 (C=O), 132.79, 131.87, 128.63, 128.47, 128.37, 127.60, 125.23, 121.36



¹H NMR 4,5-dichloroanthracene-9-carboxylic acid in DMSO-d₆



¹³C NMR 4,5-dichloroanthracene-9-carboxylic acid in DMSO-d₆

Synthesis References

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S4: Crystallographic information for 4,5diF-9AC:

Procedure:

A light yellow thin needle fragment (0.475 x 0.032 x 0.010 mm³) was used for the single crystal x-ray diffraction study of $C_{15}H_8F_2O_2$ (sample cb71_0m). The crystal was coated with paratone oil and mounted on to a cryo-loop glass fiber. X-ray intensity data were collected at 100(2) K on a Bruker APEX2 (ref. 1) platform-CCD x-ray diffractometer system (fine focus Mo-radiation, $\lambda = 0.71073$ Å, 50KV/30mA power). The CCD detector was placed at a distance of 5.0600 cm from the crystal.

A total of 3600 frames were collected for a hemisphere of reflections (with scan width of 0.3° in ω and ϕ , starting ω and 2 θ angles of -30° , and ϕ angles of 0°, 90°, 180°, and 270° for every 600 frames, and 1200 frames with ϕ -scan from 0°-360°, 180 sec/frame exposure time). The frames were integrated using the Bruker SAINT software package (**ref. 2**) and using a narrow-frame integration algorithm. Based on a monoclinic crystal system, the integrated frames yielded a total of 13979 reflections at a maximum 2 θ angle of 50.698° (0.83 Å resolution), of which 1979 were independent reflections ($R_{int} = 0.0976$, $R_{sig} = 0.0591$, redundancy = 7.1, completeness = 100%) and 1318 (66.6%) reflections were greater than 2σ (I). The unit cell parameters were, **a** = 3.7462(11) Å, **b** = 9.5568(27) Å, **c** = 29.9042(84) Å, β = 90.3574(47)°, V = 1072.6(5) Å³, Z = 4, calculated density $D_c = 1.602$ g/cm³. Absorption corrections were applied (absorption coefficient μ = 0.129 mm⁻¹, min/max transmission = 0.941/0.999) to the raw intensity data using the SADABS program (**ref. 3**).

The Bruker SHELXTL software package (**ref. 4**) was used for phase determination and structure refinement. The distribution of intensities ($E^{2}-1 = 1.006$) and systematic absent reflections indicated one possible space group, P2(1)/n. The space group P2(1)/n (#14) was later determined to be correct. Direct methods of phase determination followed by two Fourier cycles of refinement led to an electron density map from which most of the non-hydrogen atoms were identified in the asymmetric unit of the unit cell. With subsequent isotropic refinement, all of the non-hydrogen atoms were identified. There was one molecule of $C_{15}H_8F_2O_2$ present in the asymmetric unit of the unit cell. The intermolecular hydgrogen bond distances and angles are given in Table 7.

Atomic coordinates, isotropic and anisotropic displacement parameters of all the non-hydrogen atoms were refined by means of a full matrix least-squares procedure on F^2 . The H-atoms were included in the refinement in calculated positions riding on the atoms to which they were attached, except the OH hydrogen atom involved in intermolecular hydrogen bonding was refined unrestrained. The refinement converged at R1 = 0.0478, wR2 = 0.1117, with intensity I>2s (I). The largest peak/hole in the final difference map was 0.283/-0.334 e/Å³.

Table 1. Crystal data and structure refinement for 4,5diF-9AC.		
Identification code	cb71_0m	
Empirical formula	C15 H8 F2 O2	
Formula weight	258.21	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 3.7462(11) Å	a= 90°.

	b = 9.557(3) Å	b= 90.357(5)°.
	c = 29.904(8) Å	g = 90°.
Volume	1070.6(5) Å ³	
Ζ	4	
Density (calculated)	1.602 Mg/m ³	
Absorption coefficient	0.129 mm ⁻¹	
F(000)	528	
Crystal size	$0.475 \ x \ 0.032 \ x \ 0.010 \ mm^3$	
Theta range for data collection	1.362 to 25.349°.	
Index ranges	-4<=h<=4, -11<=k<=11, -36	5<=l<=36
Reflections collected	13979	
Independent reflections	1979 [R(int) = 0.0976]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equiva	lents
Refinement method	Full-matrix least-squares on	F ²
Data / restraints / parameters	1979 / 0 / 173	
Goodness-of-fit on F ²	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0478, wR2 = 0.1117	
R indices (all data)	R1 = 0.0856, wR2 = 0.1273	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.283 and -0.334 e.Å ⁻³	

S5: Crystallographic information for 4,5diCl-9AC:

Procedure:

A light-yellow thin needle fragment (0.500 x 0.042 x 0.032 mm³) was used for the single crystal x-ray diffraction study of $C_{15}H_8Cl_2O_2$ (sample cb72_0m). The crystal was coated with paratone oil and mounted on to a cryo-loop glass fiber. X-ray intensity data were collected at 100(2) K on a Bruker APEX2 (ref. 1) platform-CCD x-ray diffractometer system (fine focus Mo-radiation, 1 = 0.71073 Å, 50KV/30mA power). The CCD detector was placed at a distance of 5.0600 cm from the crystal.

A total of 4800 frames were collected for a sphere of reflections (with scan width of 0.3° in w and f, starting w and 2q angles of -30° , and f angles of 0° , 90° , 120° , 180° , 240° , and 270° for every 600 frames, and 1200 frames with f-scan from 0° -360°, 60 sec/frame exposure time). The frames were integrated using the Bruker SAINT software package (**ref. 2**) and using a narrow-frame integration algorithm. Based on a monoclinic crystal system, the integrated frames yielded a total of 30670 reflections at a maximum 2q angle of 60.068° (0.71 Å resolution), of which 3482 were independent reflections ($R_{int} = 0.0692$, $R_{sig} = 0.0388$, redundancy = 8.8, completeness = 100%) and 2910 (83.6%) reflections were greater than 2s(I). The unit cell parameters

were, $\mathbf{a} = 18.665(2)$ Å, $\mathbf{b} = 3.8338(4)$ Å, $\mathbf{c} = 33.213(4)$ Å, $\mathbf{b} = 95.3374(17)^{\circ}$, V = 2366.4(4) Å³, Z = 8, calculated density $D_c = 1.621$ g/cm³. Absorption corrections were applied (absorption coefficient m = 0.524 mm⁻¹; min/max transmission = 0.779/0.983) to the raw intensity data using the SADABS program (**ref. 3**).

The Bruker SHELXTL software package (**ref. 4**) was used for phase determination and structure refinement. The distribution of intensities ($E^{2}-1 = 1.001$) and systematic absent reflections indicated one possible space group, C2/c. The space group C2/c (#15) was later determined to be correct. Direct methods of phase determination followed by two Fourier cycles of refinement led to an electron density map from which most of the non-hydrogen atoms were identified in the asymmetric unit of the unit cell. With subsequent isotropic refinement, all of the non-hydrogen atoms were identified. There was one disordered molecule of $C_{15}H_8Cl_2O_2$ present in the asymmetric unit of the unit cell. The Cl-atoms bonded to C4 and C5 were modeled with disordered. The disordered Cl/H site occupancy ratio was 96%/4%. The intermolecular hydrogen bond distances and angle are given in Table 7.

Atomic coordinates, isotropic and anisotropic displacement parameters of all the non-hydrogen atoms were refined by means of a full matrix least-squares procedure on F^2 . The H-atoms were included in the refinement in calculated positions riding on the atoms to which they were attached, except the OH hydrogen atom involved in intermolecular hydrogen bonding was refined unrestrained. The refinement converged at R1 = 0.0443, wR2 = 0.1126, with intensity I>2s (I). The largest peak/hole in the final difference map was 0.938/-0.389 e/Å³.

Identification code	4,5diCl-9AC	
Empirical formula	C15 H8.07 Cl1.93 O2	
Formula weight	288.70	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 18.665(2) Å	a= 90°.
	b = 3.8338(4) Å	b=95.3374(17)°.
	c = 33.213(4) Å	g = 90°.
Volume	2366.4(4) Å ³	
Ζ	8	
Density (calculated)	1.621 Mg/m ³	
Absorption coefficient	0.524 mm ⁻¹	
F(000)	1175	
Crystal size	0.500 x 0.042 x 0.032 mm ³	
Theta range for data collection	2.192 to 30.034°.	
Index ranges	-26<=h<=26, -5<=k<=5, -46<=l<=46	
Reflections collected	30670	
Independent reflections	3482 [R(int) = 0.0692]	

Table 2. Crystal data and structure refinement for 4,5diCl-9AC.

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Completeness to theta = 25.242°	100.0 %
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3482 / 0 / 177
Goodness-of-fit on F ²	1.066
Final R indices [I>2sigma(I)]	R1 = 0.0443, wR2 = 0.1126
R indices (all data)	R1 = 0.0546, wR2 = 0.1199
Extinction coefficient	n/a
Largest diff. peak and hole	0.938 and -0.389 e.Å ⁻³

X-ray References

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- 4. SHELXTL, version 2013/4, Bruker (2013), Bruker AXS Inc., Madison, Wisconsin, USA.