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Hydrogen and Halogen Bonding in the Crystal Structure of a 1,3,5-Substituted 2,4,6-Triethylbenzene Consisting of Three Phenanthroline Units

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1,3,5-Tris-[*N*-(1,10-phenanthrolin-2-yl-carbonyl)aminomethyl]-2,4,6-triethylbenzene (**1**) was synthesized and its crystal structure was examined. X-ray structural analysis revealed the presence of hydrogen-bonded water molecules as well as hydrogen- and halogen-bonded chloroform molecules in the crystal structure of **1**. The $\mathbf{1}\cdot H_2O\cdot CHCl_3$ associate is stabilized by N–H···O, O–H···N, O–H···O, C–H···O, and C–H···Cl hydrogen bonds as well as C–Cl···O, C–Cl···π, and C–Cl···Cl interactions. Particularly short C–H···O hydrogen bonds with H···O and C···O distances of 1.89 and 2.94 Å, respectively, are observed in the crystal structure. The H···O distance is approximately 30 % shorter than the sum of the van der Waals radii of involved atoms.

Introduction

Hydrogen bonding is by far the most frequently occurring noncovalent interaction in supramolecular and biological recognition processes.[1] Various types of hydrogen bonds, including weak hydrogen bonds based on C–H donors,[2–7] have been utilized for the formation of supramolecular systems. Recent reports[8] have highlighted the efficacy of a less widely exploited noncovalent interaction for the generation of supramolecular architectures, namely, the halogen-bonding interaction. The term halogen bonding is used in the literature[8a–8c] for defining any noncovalent interaction involving halogen atoms as electron acceptors (as halogen-bond donors). Halogen bonds have been shown to be of great importance in crystal engineering, material chemistry (liquid crystals, magnetic and conducting materials), and biological systems.[8a–8c]

In this contribution, we describe both hydrogen- and halogen-bonding interactions observed in the crystal structure of 1,3,5-tris-[*N*-(1,10-phenanthrolin-2-yl-carbonyl)-aminomethyl]-2,4,6-triethylbenzene (**1**). X-ray structural analysis revealed the presence of hydrogen-bonded water molecules as well as hydrogen- and halogen-bonded chloroform molecules in the crystal structure of phenanthroline-based compound **1**.

Results and Discussion

The synthesis of compound **1** is outlined in Scheme 1. The synthesis started from 1,3,5-tris(bromomethyl)-2,4,6-triethylbenzene (**2**),[9] which was converted into compound **3** through reaction with potassium phthalimide. Treatment of **3** with hydrazine gave 1,3,5-tris(aminomethyl)-2,4,6-triethylbenzene (**4**), which was converted into compound **1** through a reaction with 1,10-phenanthroline-2-carbonyl chloride (**5**). Carbonyl chloride **5** was prepared by reaction of 1,10-phenanthroline-2-carboxylic acid[10] with thionyl chloride.

Single-crystal X-ray analysis of **1** revealed the presence of three water molecules in the cavity of **1**, as shown in Figure 1 (crystals were grown by slow evaporation of the solvent from a chloroform solution of compound **1**); aggregate $\mathbf{1}\cdot 3H_2O$ is stabilized by NH···O, OH···N, and OH···O hydrogen bonds (see Table 1). Each water molecule donates a hydrogen bond to N-10 of phenanthroline and accepts a hydrogen bond from the amide. In addition, OH···N-1-phenanthroline (secondary) hydrogen bonds (the O–H group is involved in an asymmetric three-center hydrogen-bond system) can be observed in the crystal structure (for a discussion on the secondary electrostatic interaction model, see ref.[11]; for a description of three-center hydrogen bonds, see ref.[12]). It should be noted that the three phenanthroline groups of **1** point to the same face of the central phenyl ring, whereas the three ethyl groups point in the opposite direction.

The chloroform molecules are located outside the cavity of compound **1** and are involved in Cl_3C–H···O=C, Cl_2HC–Cl···O=C, Cl_2HC–Cl···phenyl, Cl_3C–H···Cl–$CHCl_2$, and Cl_2HC–Cl···Cl–$CHCl_2$ interactions, as shown in Figures 2 and 3 (see also Table 2). The C–H···O hydrogen bonds in-

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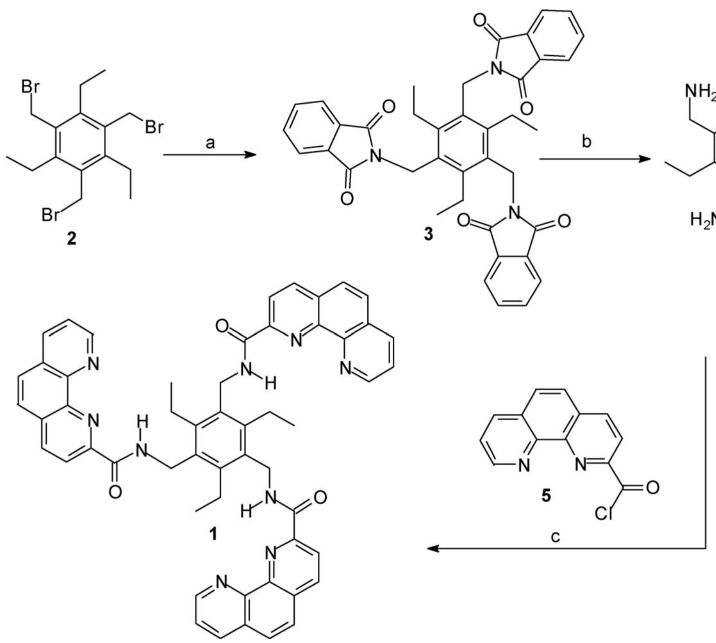

Scheme 1. Synthesis of compound **1**: Reagents and conditions: (a) potassium phthalimide, DMSO; (b) hydrazine hydrate, ethanol/toluene; (c) NEt_3, THF/CH_2Cl_2.

volving the C–H unit of chloroform and the carbonyl oxygen atom of **1** are shown in Figure 2a. There are two C–H···O interactions with H···O distances of 1.89 Å (interaction a, C···O distance 2.94 Å) and 2.15 Å (interaction b, C···O distance 3.11 Å). The first is approximately 30% shorter than the sum of the van der Waals radii of involved atoms (2.72 Å[13]). This intermolecular hydrogen bond is significantly shorter than that reported in the literature for the Cl_3C–H···O system (the mean H···O and C···O separations were found to be 2.22 and 3.16 Å, respectively[14]).

Examples of Cl_2HC–Cl···O=C halogen bonds[15] are shown in Figure 2a. The chlorine atom is engaged in the formation of a C–Cl···O halogen bond (*n*-type halogen bonding, interaction c) with Cl···O distance of 3.09 Å (C···O distance 4.82 Å, C–Cl···O angle 167°). The Cl···O distance is approximately 5% shorter than the sum of the van der Waals radii of involved atoms (3.27 Å[13]). Chlorine atoms not involved in *n*-type halogen bonding or in hydrogen bonding participate in π-type halogen bonding with the phenyl ring of **1** (see Figures 2b and 3). The distances of the chlorine atoms from the center of the phenyl ring of **1** are 3.15 Å (the Cl···C_{phenyl} separations vary from 3.29 to 3.58 Å, whereas angles vary from 138 to 170°; see Table 2). As demonstrated by Legon,[16] halogen bonding involving *n*-type electron donors (such as oxygen or nitrogen) and π-type electron donors (double and triple bonds, aromatic groups) follow the same geometric and energetic rules; however, π-type electron donors are weaker than *n*-type donors. As reported in the literature,[17] the X-ray structural data for the intermolecular associates between a chlorocarbon and neutral aromatic donors are rather rare. Most frequently, the halocarbons show over-the-rim π-bonding (over-the-rim coordination to the aromatic C–C bond), although coordination over the ring center (as in the case of $CHCl_3\cdot$**1**) and over individual carbon atoms is also observed.[17]

The phenanthroline CH groups in the 3-, 6-, and 8-positions of the heterocyclic ring participate in C–H···Cl hydrogen bonds (for an example of a phenanthroline–CH···Cl–$CHCl_2$ interaction, see Figure 3). The H···Cl distances are in the range of 2.61–2.93 Å and the C···Cl distances lie between 3.68 and 4.01 Å (the C–H···Cl angles are in the range of 171–178°, see Table 2).

Between the chloroform molecules, short C–H···Cl and C–Cl···Cl interactions are observed. The Cl_3C–H···Cl–$CHCl_2$ hydrogen bonds show H···Cl distances of 2.89 Å (C···Cl, 3.69 Å), whereas the Cl···Cl distances of the Cl_2HC–Cl···Cl–$CHCl_2$ interactions amount to 3.30 Å (C···Cl, 4.51 and 5.03 Å; C–Cl···Cl angles 123 and 171°; see Table 2). It should be noted that the definition halogen bonding comprises also halogen···halogen interactions,[8a,15a] which were not included in the early meaning of the term.

Conclusions

In this investigation, 1,3,5-tris-[*N*-(1,10-phenanthrolin-2-yl-carbonyl)aminomethyl]-2,4,6-triethylbenzene (**1**) was synthesized and its crystal structure was examined. X-ray structural analysis revealed the presence of hydrogen-bonded water molecules as well as hydrogen- and halogen-bonded[15] chloroform molecules in the crystal structure of **1**. Three water molecules are present in the cavity of **1**, whereas the chloroform molecules are located outside the

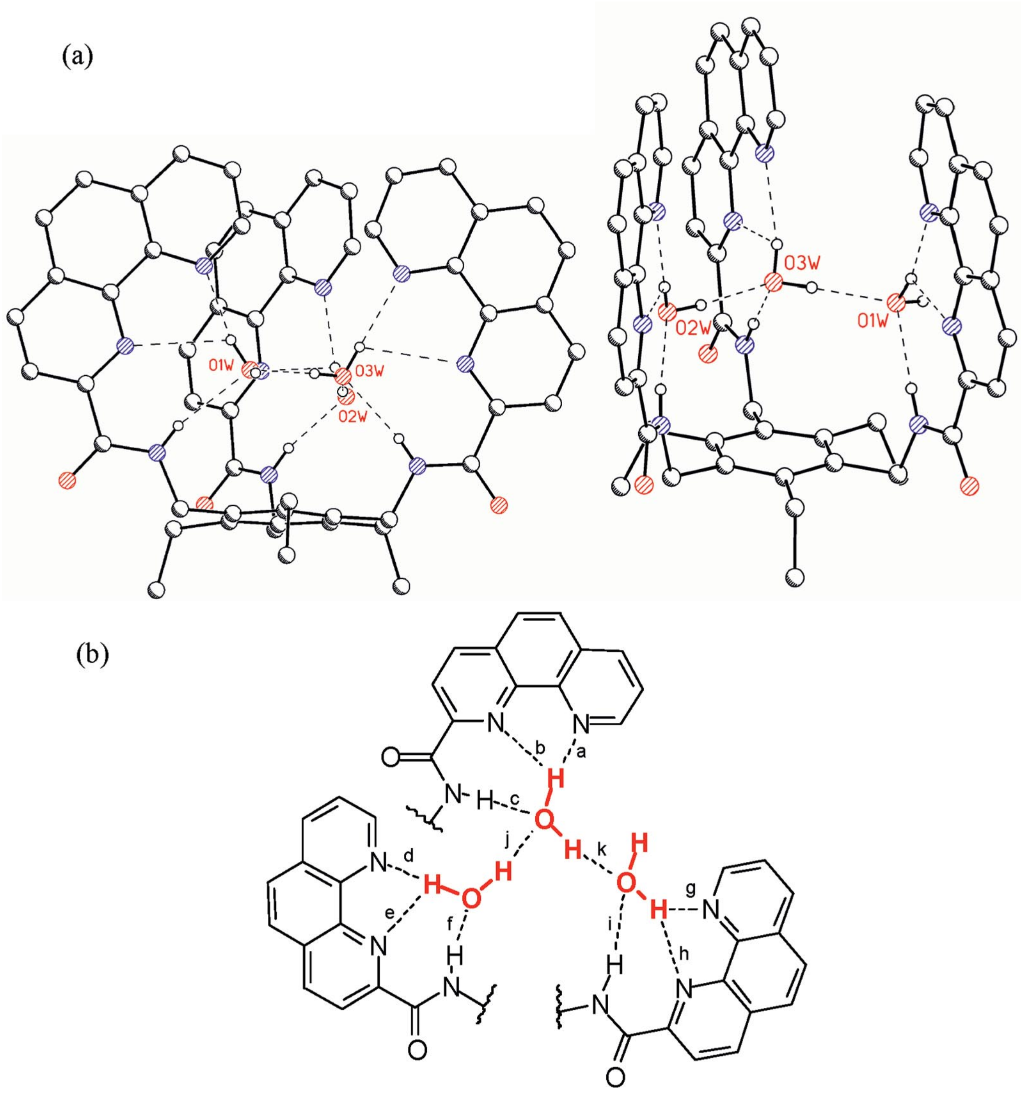

Figure 1. (a) Molecular structure of **1** with hydrogen-bonded water molecules in the crystal (two different representations, C–H bonds are omitted for clarity). (b) Schematic representation of the binding motifs in the cavity of **1** (the a–k contact distances are given in Table 1).

cavity. The associate **1**·water·chloroform is stabilized by N–H···O, O–H···N, O–H···O, C–H···O, and C–H···Cl hydrogen bonds as well as C–Cl···O, C–Cl···π, and C–Cl···Cl interactions. Particularly short C–H···O hydrogen bonds with H···O and C···O distances of 1.89 and 2.94 Å, respectively, can be observed in the crystal structure. The H···O distance is approximately 30% shorter than the sum of the van der Waals radii of the involved atoms.

It should be also noted that phenanthroline-based compounds have been used as synthetic receptors for diols[18] and carbohydrates.[19] Our previous studies showed that phenanthroline-based receptors[19] are particularly powerful receptors for carbohydrates, having the rare capability to extract sugars from water into nonpolar organic solutions. The analysis of the binding properties of compound **1** towards carbohydrates is under investigation.

Table 1. X–H···Y distances and angles for **1**·3H_2O (X, Y = O or N).

XH···Y interaction (see Figure 1)	XH···Y [Å]	X···Y [Å]	X–H···Y angle [°]
HOH···N10-phenanthroline (a)	2.03	2.88	166
HOH···N1-phenanthroline (b)[a]	2.67	3.13	114
H_2O···HNCO (c)	2.12	2.95	171
HOH···N10-phenanthroline (d)	2.10	2.93	166
HOH···N1-phenanthroline (e)[a]	2.60	3.13	122
H_2O···HNCO (f)	2.10	2.92	163
HOH···N10-phenanthroline (g)	1.91	2.76	166
HOH···N1-phenanthroline (h)[a]	2.65	3.16	120
H_2O···HNCO (i)	2.28	3.08	161
H_2O···HOH (j)	1.98	2.82	174
H_2O···HOH (k)	1.93	2.82	178

[a] Weaker branch of an asymmetric three-center hydrogen-bond system.

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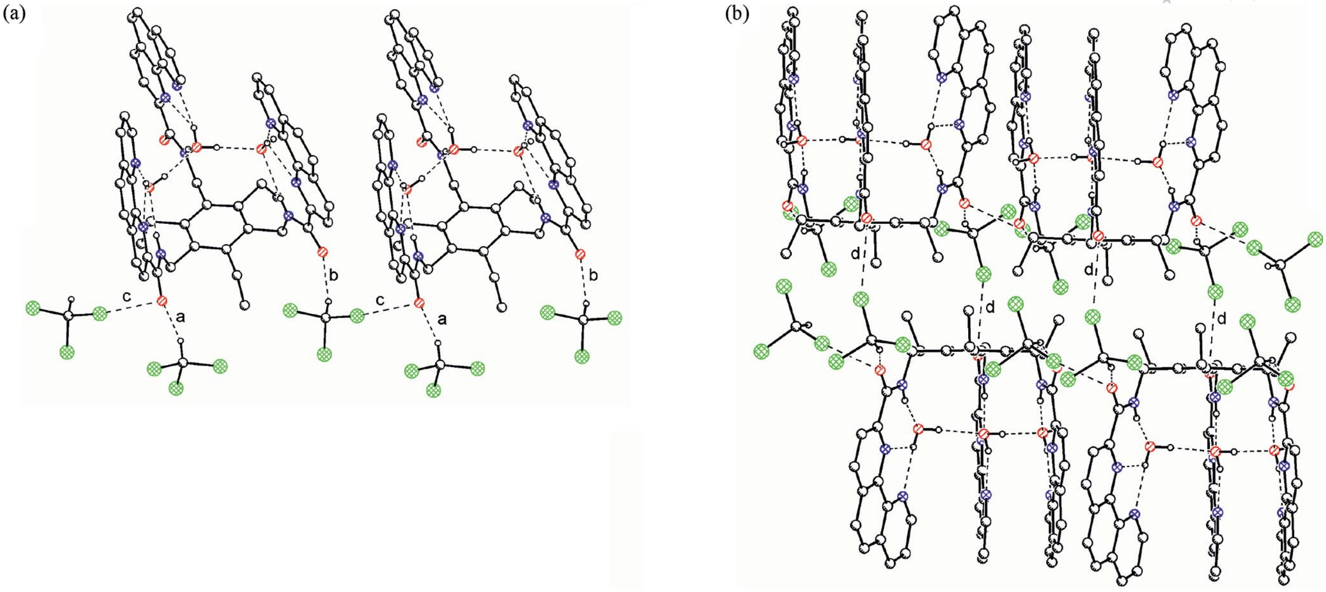

Figure 2. (a) CH···O hydrogen bonds (interactions a and b) and CCl···O halogen bonds (interaction c) in the crystal structure of **1** (the contact distances are given in Table 2; C–H bonds of **1** are omitted for clarity). (b) Packing of the molecules in the crystal of **1** (dotted lines represent hydrogen and halogen bonds; C–H bonds of **1** are omitted for clarity); the contact d represents CCl···Ph–center π-type halogen bond (the contact distance is given in Table 2).

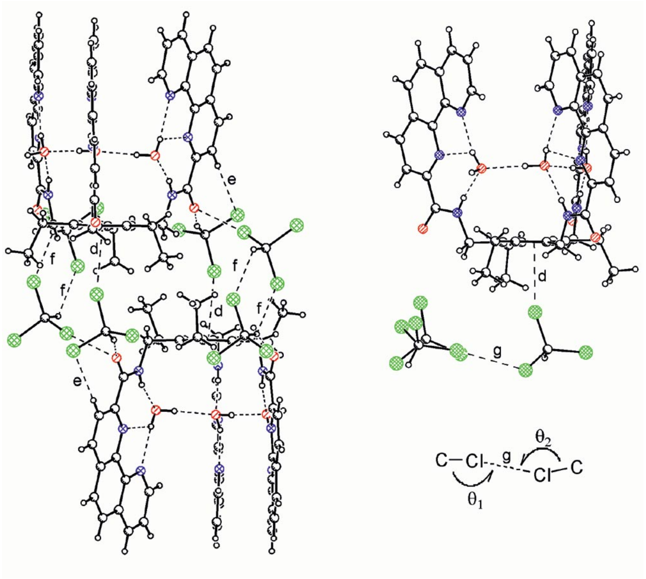

Figure 3. CCl···Ph–center (contact d), phenanthroline–CH···ClCHCl$_2$ (contact e), Cl$_3$CH···ClCHCl$_2$ (contact f), and Cl$_2$HCCl···ClCHCl$_2$ (contact g) interactions in the crystal structure of **1** (the contact distances are given in Table 2; for θ_1 and θ_2 angles, see Table 2).

Experimental Section

1,3,5-Tris(phthalimidomethyl)-2,4,6-triethylbenzene (3): A mixture of 1,3,5-tris(bromomethyl)-2,4,6-triethylbenzene (**2**; 10.0 g, 22.7 mmol) and potassium phthalimide (16.8 g, 90.8 mmol) in dry dimethyl sulfoxide (150 mL) was heated to 100 °C for 8 h. After the mixture was cooled to room temperature, water was added (200 mL) and the formed precipitate was filtered and washed with water (300 mL). Then, the precipitate was suspended in water (150 mL), and the suspension was extracted with CHCl$_3$ (3 × 100 mL). The combined organic layer was washed with water, dried with MgSO$_4$, and concentrated. Yield: 13.5 g (90 %). M.p. 228–229 °C. R_f = 0.51 (toluene/ethyl acetate, 3:1). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 1.10 (t, J = 7.4 Hz, 9 H), 3.09 (q, J = 7.4 Hz, 6 H), 4.93 (s, 6 H), 7.67–7.70 (m, 6 H), 7.81–7.83 (m, 6 H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$): δ = 15.7, 23.3, 37.5, 123.2, 129.5, 132.0, 133.9, 145.5, 168.2 ppm. HRMS (ESI): calcd. for C$_{39}$H$_{33}$N$_3$O$_6$Na 662.2267; found 662.2262.

1,3,5-Tris-(aminomethyl)-2,4,6-triethylbenzene (4):[9] Compound **3** (9.54 g, 14.9 mmol) was dissolved in a mixture of dry ethanol/toluene (2:1) and heated at reflux with hydrazine hydrate (3 mL, 93 mmol) for 20 h. Afterwards, the reaction mixture was filtered, the precipitate was suspended in an aqueous solution of KOH (40 %, 150 mL), and the suspension was extracted with chloroform (3 × 150 mL). The combined organic extract was washed with water, dried with MgSO$_4$, filtered, and concentrated. Yield: 3.45 g (93 %). M.p. 137–139 °C. R_f = 0.10 (chloroform/methanol, 7:1). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 1.23 (t, J = 7.5 Hz, 9 H), 1.35 (br. s, 6 H), 2.82 (q, J = 7.5 Hz, 6 H), 3.37 (br. s, 6 H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$): δ = 16.1, 22.7, 36.9, 131.4, 143.9 ppm.

1,3,5-Tris-[*N*-(1,10-phenanthrolin-2-yl-carbonyl)aminomethyl]-2,4,6-triethylbenzene (1)

(a) Synthesis of 5: A mixture of 1,10-phenanthroline-2-carboxylic acid[10] (0.48 g, 2.14 mmol) and thionyl chloride (20 mL) was heated at reflux for 6 h. Thionyl chloride was then removed in vacuo. Afterwards, THF (3 × 20 mL) was added and the solvent was removed in vacuo. The crude product was used directly for further reaction.

(b) Synthesis of 1: A solution of **5** in THF/CH$_2$Cl$_2$ (1:2, 60 mL) was added dropwise to a solution of 1,3,5-triaminomethyl-2,4,6-triethylbenzene (**4**; 0.15 g, 0.60 mmol) and triethylamine (0.25 mL, 1.80 mmol) in CH$_2$Cl$_2$ (20 mL). After complete addition, the mixture was stirred at room temperature for 24 h. Afterwards, the or-

Table 2. C–H···Y and C–Cl···Y distances and angles in the crystal structures of **1**·H_2O·$CHCl_3$ (Y = O, Cl, C_{phenyl} or Ph–center).

C–H···Y/C–Cl···Y interaction	CH···Y/CCl···Y [Å]	C···Y [Å]	C–H···Y/C–Cl···Y angle [°]	Figure[f]
Cl_3C–H···O=C (a)[a]	1.89	2.94	161	Figure 2a
Cl_3C–H···O=C (b)[a]	2.15	3.11	148	Figure 2a
Cl_2HC–Cl···O=C (c)[a]	3.09	4.82	167	Figure 2a
Cl_2HC–Cl···Ph-center (d)[a]	3.15	4.82	161	Figures 2b, 3
Cl_2HC–Cl···C_{phenyl}	3.29–3.58	4.85–5.18	138–170	
phenanthroline-C3–H···Cl–$CHCl_2$ (e)[a]	2.93	4.01	178	Figure 3
phenanthroline-C6–H···Cl–$CHCl_2$	2.87	3.91	172	
phenanthroline-C8–H···Cl–$CHCl_2$	2.61	3.68	171	
Cl_3C–H···Cl–$CHCl_2$ (f)[a]	2.89	3.69	132	
Cl_2HC^A–Cl^A···Cl^B–C^BHCl_2 (g)[a]	3.30	5.03[b]	171[d]	Figure 3
		4.51[c]	123[e]	Figure 3

[a] Noncovalent interaction shown in Figures 2 and 3. [b] C^A···Cl^B distance (C–H distances normalized to 1.08 Å). [c] Cl^A···C^B distance. [d] θ_1 (C^A–Cl^A···Cl^B angle), see Figure 3. [e] θ_2 (Cl^A···Cl^B–C^B angle). [f] Two of the three chloroform molecules are slightly disordered (ca. 86:14). Packing diagrams and discussion are based only on the major components.

ganic solvents were removed in vacuo, and the resulting solid was purified by column chromatography (chloroform/methanol, 10:1). Yield: 0.36 g (68%). M.p. 151–152 °C. R_f = 0.58 (silica gel; chloroform/methanol, 10:1). ^1H NMR (400 MHz, $CDCl_3$): δ = 1.40 (t, J = 7.5 Hz, 9 H), 3.07 (q, J = 7.5 Hz, 6 H), 4.92 (d, J = 4.3 Hz, 6 H), 6.83 (dd, J = 8.1, 4.4 Hz, 3 H), 7.75 (d, J = 8.8 Hz, 3 H), 7.81 (d, J = 8.8 Hz, 3 H), 7.83 (dd, J = 4.4, 1.5 Hz, 3 H), 8.07 (dd, J = 8.1, 1.5 Hz, 3 H), 8.34 (d, J = 8.3 Hz, 3 H), 8.61 (d, J = 8.3 Hz, 3 H), 10.03 (t, J = 3.8 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 16.0, 23.6, 38.6, 122.1, 122.4, 126.9, 127.1, 128.7, 129.1, 131.4, 136.2, 137.2, 144.1, 144.7, 145.2, 148.7, 150.8, 164.7 ppm. HRMS (ESI): calcd. for $C_{54}H_{45}N_9O_3Na$ 890.3543; found 890.3539.

CCDC-739268 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgments

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