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Synthesis and reactivity of bis(cycloamidinium-2-yl)alkane bromide tribromides, brominating agents and tectons for molecular engineering

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Abstract Bis(cycloamidine-2-yl)alkanes easily form bromide tribromide salts in the reduction-oxidation processes with bromine. Bis(tetrahydroimidazolium-2-yl)ethane and bis(hexahydropyrimidinium-2-yl)-ethane bromide tribromides are such new convenient brominating agents for aromatic amides in chemo- and regioselective electrophilic substitutions and α-bromination reactions.

Keywords Bis(cycloamidinum-2-yl)alkane bromide tribromides; Hydrogen sponge; Solid organic ammonium tribromide; Bromination.

Introduction

Solid organic [1–8] and ionic liquid [9–12] ammonium tribromides, equivalents of molecular bromine, found increasing application in organic synthesis as chemo-, regio-, and diasteroselective [1, 13, 14] brominating agents in the α-bromination of ketones [1, 4, 6, 15] and ketals [1, 13], electrophilic addition [1, 3, 4, 14, 16–20], and substitution [4, 7, 11, 18, 21–23]. They are also used as catalysts for the oxidation of aromatic aldehydes to carboxylic acids [24] or ω-bromoesters [25] and dialkyl and alkyl aryl sulfides to sulfoxides [5]. Bis(dialkylamide) hydrobromide perbromides are also precursors of the hypobromite ion [26], an effective α-nucleophile and potentially useful decontaminant. *n*-Tetrabutylammoniumtribromide in alcohol generates *in situ* hydrogen bromide, applied in the protection of carbonyl [27] and hydroxyl groups [28] and the cleavage of ethers and dithioacetals [29]. Solid organic ammonium tribromides catalyze also heterocyclizations, such as aziridation [30] and the synthesis of benzothiazoles [31]. Some solid organic and especially ionic liquid ammonium tribromides react under solvent free conditions [4, 9–12] or in silica sol-gel encaged systems [8].

Organic ammonium tribromides (OATB) are usually formed in the reaction of nitrogen bases with hydrobromide and bromine [1, 6–8] or bromide salts with bromine [3, 9, 10, 12]. To avoid the use of toxic molecular bromine the new synthesis protocols include generation of OATB followed by the *in situ* oxidation of organic [18, 22] or inorganic [4, 5] bromides. Organic ammonium tribromides are also derived by addition of two equivalents of bromine to nitrogen heterocycles containing double or triple bonds, when an electrophilic addition is correlated with a cyclization [32–37].

Results and discussion

In this paper we report the preparation of bis(cycloamidinium-2-yl)alkane bromide tribromides **5**, **6**, **7**, and **8** by reaction of bis(cycloamidin-2-yl)alkanes **1**, **2**, **3**, and **4** [38] with molecular bromine, where

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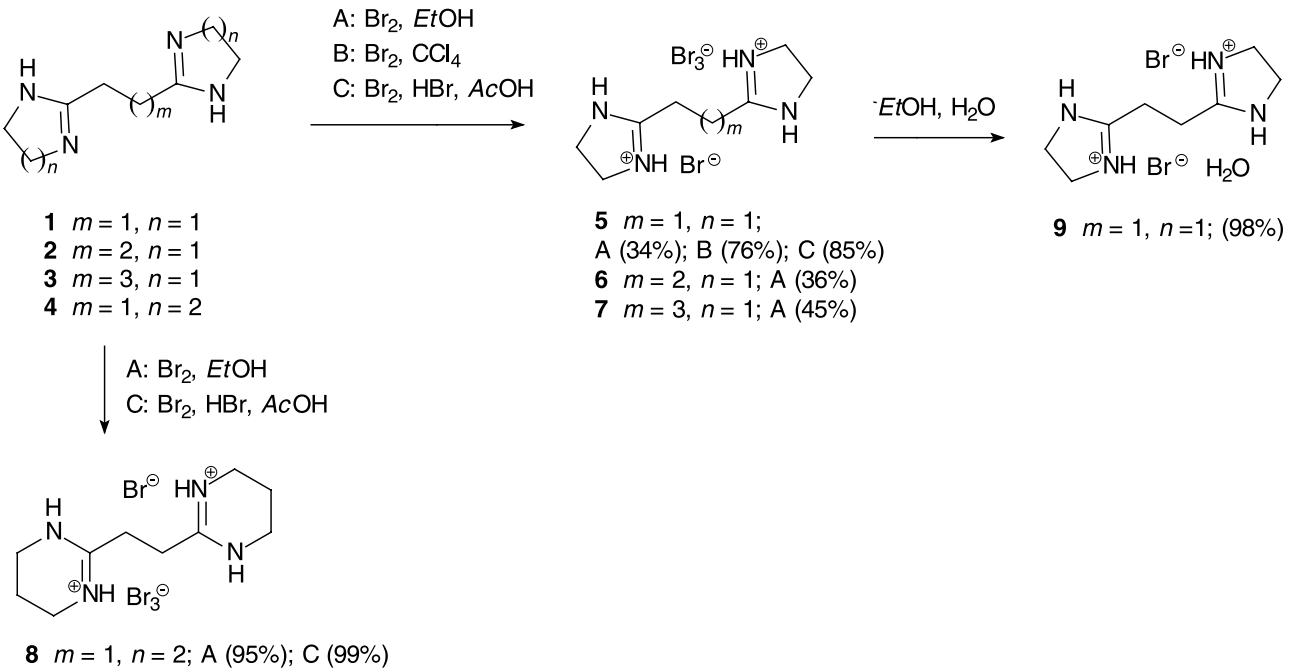


1 *m* = 1, *n* = 1
2 *m* = 2, *n* = 1
3 *m* = 3, *n* = 1
4 *m* = 1, *n* = 2

5 *m* = 1, *n* = 1;
A (34%); B (76%); C (85%)
6 *m* = 2, *n* = 1; A (36%)
7 *m* = 3, *n* = 1; A (45%)

9 *m* = 1, *n* =1; (98%)

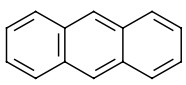
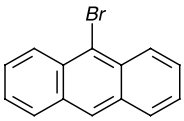
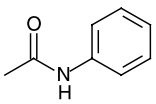
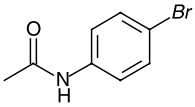
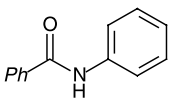
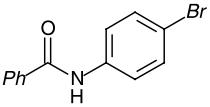
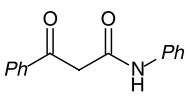
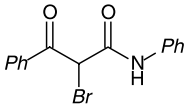
8 *m* = 1, *n* = 2; A (95%); C (99%)

Scheme 1

the formation of tribromide salts requires *in situ* reduction of bromine cations (Scheme 1). Alternatively, the bromide tribromides **5** and **8** are formed with bromine in the presence of hydrogen bromide in *Ac*OH.

We observed that the strong conjugation between imino double bond and the electron pair on the amino nitrogen limited the reactivity of bis(cycloamidin-2-yl)alkane. These strong bases with bromine in anhydrous *Et*OH or CCl_4 give exclusively the diprotonated form possessing four equivalent N–H protons and anionic bromide tribromide. These equivalents explain that bis(cycloamidin-2-yl)alk-

Table 1 Bromination of antracene and aromatic amides with **5** and **8**

Entry	Substrate	Product, mp/°C	Conditions for **5** (1 equiv.)	Yield/%; mp/°C	Conditions for **8** (1 equiv.)	Yield/%; mp/°C
1		 99 [44]	*Ac*OH, refluxed, 30 min	29; 98	*Ac*OH, refluxed, 30 min	74; 97
2		 168 [45]	50% *DMF*, rt, 30 min	99; 169	50% *DMF*, rt, 40 min	85; 169–170
3		 203–204 [46]	50% *DMF*, rt, 20 h	20; 205	50% *DMF*, rt, 20 h	97; 199–200
4		 133–134 [47]	50% *DMF*, rt, 25 h	68; 125	50% *DMF*, rt, 25 h	90; 128

anes are proton sponges and force the reduction-oxidation processes, *i.e.*, reduction of bromine cation to bromide and probably the oxidation of water to hydrogen peroxide. It was proved [39], that imidazolidines are extremely strong organic bases by the determination of pK_b value for 1,2-bis(4-methyl-4,5-dihydro-1*H*-imidazol-2-yl)ethane equal to 10.5. This was performed by potentiometric titration of the water solution of the compound by hydrochloric acid. It is interesting that the potentiometric curve shows only one point of inflexion *i.e.*, that bisimidazolidine as a strong base is protonated by water and in fact OH^- ions are titrated. We found in literature that other strong bases such as guanidine [40], oxalamidine [41], and imidazolidine [42, 43] with bromine gave the tribromide salts in the same manner.

The bromide tribromide salt **5** easily evolved elemental bromine during crystallization in aqueous *Et*OH (Scheme 1) and forms the dibromide hydrate **9**. So, the bromide tribromide salts **5** and **8** are used as a new convenient source of elemental bromine in chemo and regioselective electrophilic substitution and α-bromination reactions (Table 1).

X-Ray analyses were performed for single crystals of the tribromide salts of compounds **5** and **8** and dibromide hydrate **9**.

Bond length analysis shows that the nitrogen atoms in the heteroatomic ring of all presented crystal structures are both in sp^2 hybridization [48]. The appearing positive charge is delocalized in the N1–C1–N2 region. The N–C bond distances, however, are not exactly the same being comprised in the ranges of 1.276(7) – 1.307(7), 1.304(4) – 1.315(4), and 1.308(5) – 1.314(5) Å for the compounds **5**, **9**, and **8**. This feature was observed also for the structures of other salts of the compounds **1** and **4**,

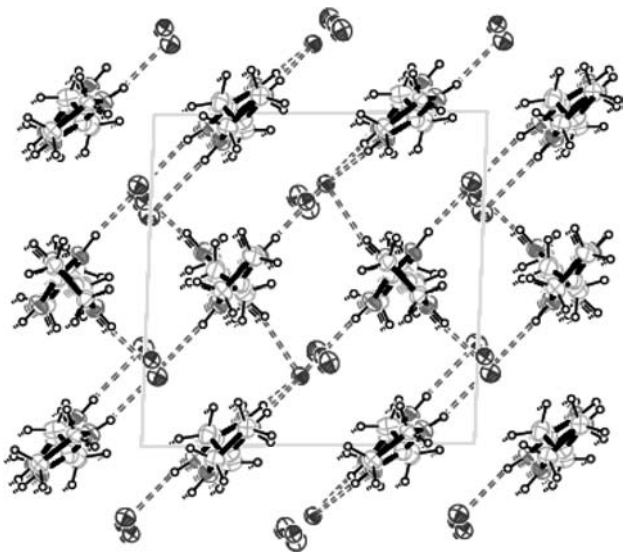


Fig. 1A Crystal packing of **5** projected along the [001] direction, showing hydrogen bond system and highly ordered aggregate of positive and negative charged "tubes". Ellipsoids are drawn at 30% probability level (visualization made with ORTEP-3.0 [56])

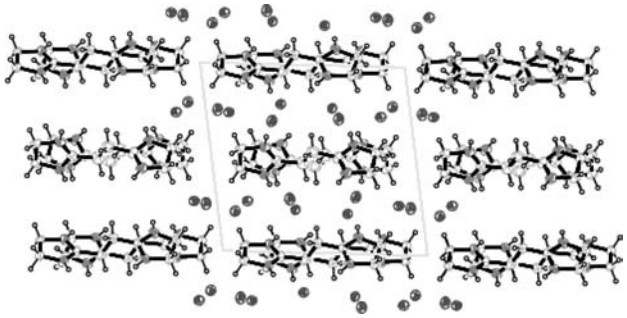


Fig. 1B Crystal packing of **5** projected along the [100] direction. Channels run along the [001] direction. Ellipsoids are drawn at 30% probability level (visualization made with ORTEP-3.0 [56])

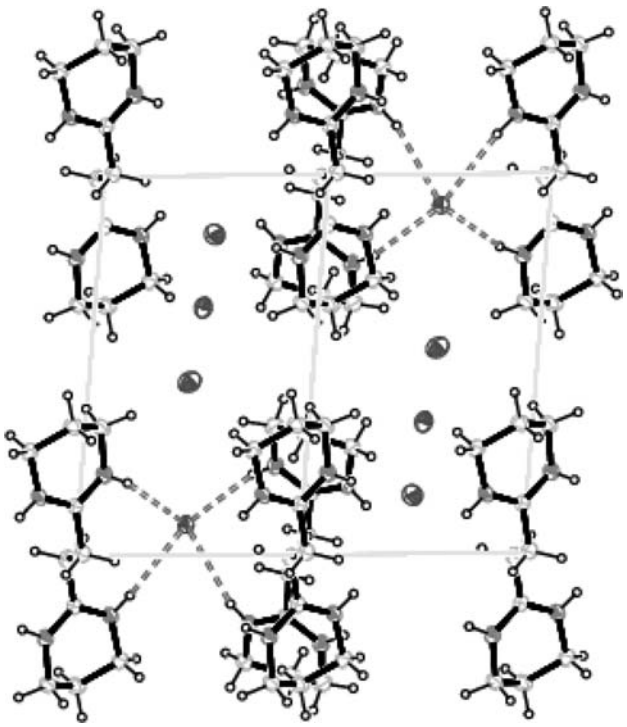


Fig. 2 Crystal packing of **8** projected along the [110] direction, showing hydrogen bond system. The highly ordered aggregate channels run along the [111] direction. Ellipsoids are drawn at 30% probability level (visualization made with ORTEP-3.0 [56])

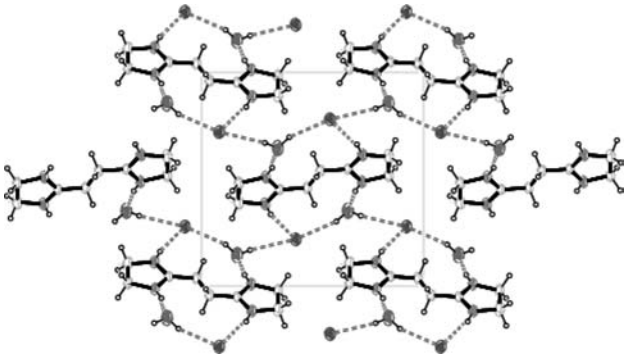


Fig. 3 Crystal packing of **9** projected along the [100] direction, showing hydrogen bond system. Water-bromide channels run along the [001] direction. Ellipsoids are drawn at 30% probability level (visualization made with ORTEP-3.0 [56])

deposited in the Cambridge Structural Database (CSD) [49], which exhibit base properties [50–53]. The differences in N–C bond lengths in the heterocyclic part are most probably connected with the different surroundings of nitrogen atoms and influence the strength of hydrogen bonds N–H···Br. The delocalization of the positive charge in the heterocyclic system of compound **5** is additionally confirmed by the short distances between the centroid (Ct) of this region and the bromine atom from tribromide ions in most cases (Ct···Br^- range 3.524(8)–3.794(8) Å), suggesting *Coulomb* interaction. In the structure **9**, these short distances are Ct···Br^- (3.354(5) Å) and, additionally, Ct···oxygen atom of the water molecule (3.315(4) Å). The difference can be noted in the structure of **8**, where the bromide anion is not centered over the N1–C1–N2 system in one molecule, while in the second molecule the Ct···Br (from Br_3^-) is longer (3.956(5) Å). The closest distance between one of the nitrogen atoms and the mentioned bromine atom is 3.557(6) Å.

In all presented structures the base molecules are highly symmetric. In all but one structure both heteroatomic rings of each molecule are mutually parallel. An alternative conformation is observed for the compound **5**. That is why in the asymmetric unit two molecules with a different geometry are present. The interplanar angle between the rings in one molecule is 11.1(2)° [54], whereas in the second molecule the conformation is much closer to perpendicular – the angle between the rings is 82.5(2)° [54]. The geometry of tribromide anions is usually close to linear with almost equal distances between bromine atoms (2.5–2.6 Å, Br–Br–Br 178°) [55].

The crystal architecture of the investigated structures is determined by hydrogen bonds N–H···Br (Figs. 1A, B, and 2). In the crystal structure of **9** O–H···Br and N–H···O interactions are present additionally (Fig. 3). Negatively charged bromide ions in **5** and **8** (Br^- and H_2O in the case of compound **9**) occur in channels, surrounded by positively charged molecules of bis(cycloamidin-2-yl)alkanes. The arrangement forms a kind of highly ordered aggregate of positively and negatively charged ''tubes'' (Fig. 1A as an example) suggesting interesting electrical properties of these tectons for molecular engineering.

Experimental

Melting points were determined on a *Boetius* PHMK 05 melting point apparatus. IR spectra: Bruker IFS 48 in KBr pellets or Nujol. NMR spectra: Bruker AMX 500 NMR (^{1}H: 500.14 MHz ^{13}C: 125.76 MHz) in *DMSO*-d_6 with *TMS* as an internal standard. Mass spectra: Finningan Mat 95 (EI, 70 eV). Microanalyses were performed with an Euro EA 3000 Elemental Analyzer; their results agreed satisfactorily with the calculated values. X-ray intensities were collected at room temperature on a Nonius Kappa CCD area detector diffractometer using MoK_α radiation ($\lambda = 0.7107$ Å) at 293(2) K.

General procedure for the preparation of bis(cycloamidinium-2-yl)alkanes bromide tribromide; 1,2-bis-(tetrahydroimidazolium-2-yl)ethane bromide tribromide (**5**, $C_8H_{16}Br_4N_4$)*, 1,2-bis(tetrahydroimidazolium-2-yl)-propane bromide tribromide* (**6**, $C_9H_{18}Br_4N_4$)*, 1,2-bis-(tetrahydroimidazolium-2-yl)butane bromide tribromide* (**7**, $C_{10}H_{20}Br_4N_4$)*, and 1,2-bis(hexahydropyrimidinium-2-yl)-ethane bromide tribromide* (**8**, $C_{10}H_{20}Br_4N_4$)

Method A: Br_2 (0.27 cm^3, 5 mmol) in 10 cm^3 anhydr. *Et*OH was added dropwise to a cooled and stirred solution of 2.6 mmol bis(cycloamidin-2-yl)alkanes **1**, **2**, **3**, or **4** in 5 cm^3 anhydr. *Et*OH. The reaction mixture was cooled in ice and stirred for additional 1 h and left overnight at rt. The precipitate was filtered and crystallized from anhydr. *Me*OH.

Method B: Br_2 (0.3 cm^3, 5.6 mmol) in 5 cm^3 anhydr. CCl_4 was added dropwise to a cooled and stirred solution of 0.475 g **1** (2.8 mmol) in 10 cm^3 anhydr. CCl_4. The reaction mixture was cooled in ice and stirred for additional 70 h and left overnight at rt. The precipitate was filtered and crystallized from anhydr. *Me*OH, yield: 0.86 g (76%).

Method C: Bis(cycloamidin-2-yl)alkanes **1** or **4** (4 mmol) was added to 12 cm^3 cooled *Ac*OH within 10 min. Then 3 cm^3 33% HBr in *Ac*OH was added gradually and after that 0.72 cm^3 Br_2 (13.4 mmol) was added dropwise to the reaction mixture. The precipitate was filtered and crystallized from anhydr. *Me*OH.

Yield for **5** (Scheme 1); mp 204–206°C; IR (KBr): $\bar{\nu} =$ 3100, 2800–2950, 1600 cm^{-1}; ^{1}H NMR (500 MHz, *DMSO*-d_6): $\delta = 2.88$ (s, 4H, CH_2–C(=N)NH), 3.82 (s, 8H, NCH_2),

10.05 (s, 4H, NH) ppm; ^{13}C NMR (500 MHz, *DMSO*-d_6): $\delta = 22.67$, 44.72, 168.728 ppm.

Crystal data for compound **5**: moiety formula $C_8H_{16}N_4^{2+} \cdot Br_3^- \cdot Br^-$, crystal size: $0.22 \times 0.13 \times 0.13$ mm^3, $M = 487.89$, triclinic, space group *P*-1, $a = 11.2595(2)$ Å, $b = 11.2715(2)$ Å, $c = 12.2573(3)$ Å, $\alpha = 81.1079(9)°$, $\beta = 80.6944(8)°$, $\gamma = 85.9162(14)°$, $V = 1514.96(5)$ Å^3, $Z = 4$, $D_c = 2.139$ g/cm^3, $\mu(\mathrm{MoK}_\alpha) = 10.61$ mm^{-1}, $F(000) = 928$; 10662 collected reflections, 6935 independent (R(int) = 0.038). The refinement parameters are: $R1 = 0.049$ for reflections with $F^2 > 2\sigma(F^2)$, $wR2 = 0.090$ (F^2), $S = 1.03$.

Yield for **6** (Scheme 1); mp 103–5°C; IR (KBr): $\bar{\nu} = 3067$, 2914, 1600 cm^{-1}; ^{1}H NMR (500 MHz, *DMSO*-d_6): $\delta = 1.854$ (m, 2H, $CH_2CH_2CH_2$), 2.55 (t, 4H, CH_2–C(=N)NH), 3.82 (s, 8H, NCH_2), 9.97 (s, 4H, NH) ppm.

Yield for **7** (Scheme 1); mp 207–209°C; IR (KBr): $\bar{\nu} =$ 3094, 2924, 1590 cm^{-1}; ^{1}H NMR (500 MHz, *DMSO*-d_6): $\delta = 2.40$ (m, 4H, CH_2CH_2), 3.67 (t, 4H, CH_2–C(=N)NH), 3.81 (s, 8H, NCH_2), 9.94 (s, 4H, NH) ppm; ^{13}C NMR (500 MHz, *DMSO*-d_6): $\delta = 24.70$, 25.74, 44.55, 170.99 ppm.

Yield for **8** (Scheme 1); mp 162°C; IR (KBr): $\bar{\nu} = 3100$, 2800–2950, 1600 cm^{-1}; ^{1}H NMR (500 MHz, *DMSO*-d_6): $\delta = 1.82$ (m, 4H, $CH_2CH_2CH_2$), 2.71 (s, 4H, CH_2–C(=N)NH), 3.31 (t, 8H, NCH_2), 9.68 (s, 4H, NH) ppm; ^{13}C NMR (500 MHz, *DMSO*-d_6): $\delta = 17.83$, 29.85, 38.59, 161.20 ppm.

Crystal data for compound **8**: moiety formula $C_{10}H_{20}N_4^{2+} \cdot Br_3^- \cdot Br^-$, crystal size: $0.25 \times 0.13 \times 0.04$ mm^3, $M = 515.94$, triclinic, space group *P*-1, $a = 8.7230(2)$ Å, $b = 8.7909(2)$ Å, $c = 11.2888(3)$ Å, $\alpha = 83.3225(8)°$, $\beta = 89.1653(8)°$, $\gamma = 82.6119(12)°$, $V = 852.65(4)$ Å^3, $Z = 2$, $D_c = 2.01$ g/cm^3, $\mu(\mathrm{MoK}_\alpha) = 9.43$ mm^{-1}, $F(000) = 496$; 7519 collected reflections, 3891 independent (R(int) = 0.027). The refinement parameters are: $R1 = 0.036$ for reflections with $F^2 > 2\sigma(F^2)$, $wR2 = 0.089$, $S = 1.04$.

Procedure for the preparation of 1,2-bis(tetrahydroimidazolium-2-yl)ethane dibromide hydrate (**9**, $C_8H_{18}Br_2N_4O$)
Compound **9** was obtained after recrystallization of **5** from ethanol solution *via* slow evaporation of the solvent in the room temperature (Scheme 1) and in the reaction of **1** (1 equiv.) with Br_2 (1 equiv.), according to the method A. Yield for **9** (46%); mp 114–117°C; IR (KBr): $\bar{\nu} = 3360$, 3120, 2966, 1600 cm^{-1}; ^{1}H NMR (500 MHz, *DMSO*-d_6): $\delta = 2.89$ (s, 4H, CH_2–C(=N)NH), 3.82 (s, 8H, NCH_2), 10.07 (s, 4H, NH) ppm; ^{13}C NMR (500 MHz, *DMSO*-d_6): $\delta = 22.66$, 44.72, 168.76 ppm.

Crystal data for compound **9**: moiety formula $C_8H_{16}N_4^{2+} \cdot 2Br^- \cdot 2H_2O$, crystal size: $0.1 \times 0.05 \times 0.03$ mm^3, $M =$ 364.08, monolinic, space group $P2_1/c$, $a = 5.4587(2)$ Å, $b = 11.3432(4)$ Å, $c = 11.6883(4)$ Å, $\beta = 93.0696(14)°$, $V =$ 722.70(4) Å^3, $Z = 2$, $D_c = 1.673$ g/cm^3, $\mu(\mathrm{MoK}_\alpha) =$ 5.60 mm^{-1}, $F(000) = 364$; 3142 collected reflections, 1621 independent (R(int) = 0.030). The refinement parameters are: $R1 = 0.038$ for reflections with $F^2 > 2\sigma(F^2)$, $wR2 = 0.079$ (F^2), $S = 1.09$. The crystals of compounds **5** and **8** were transparent, of intensive orange color, and stable in the air. The phase problem was solved with direct methods with the SHELXS-97 [57] for structures of compounds **5** and **8** and with SIR92 [58] for **9**. All structures were refined by full-matrix least-squares on F^2 (SHELXL-97) [57]. All non-hydrogen atoms were refined anisotropically. The positions of hydrogen atoms bonded to carbon atoms were calculated while those bonded to nitrogen atoms and to oxygen of water molecule were found in the difference *Fourier* map. The hydrogen atoms were refined with isotropic displacement parameters equal 1.2 or 1.5 times that of the parent atoms with the use of the riding model. Atomic coordinates, displacement parameters and all bond lengths, bond angles, as well as the lists of structure factors have been deposited with Cambridge Crystallographic Data Center (deposition numbers: compound **5**: CCDC 644373; compound **9**: CCDC 644374; compound **8**: CCDC 644372.

Procedure for the bromination of anthracene
Bromination agent **5** or **8** (3.1 mmol) was added to a solution of 0.31 g anthracene (2 mmol) in 5 cm^3 *Ac*OH within 10 min. The mixture was gently refluxed for 30 min. The cold reaction mixture was diluted with water and extracted with ether. The organic layer was separated, washed with water and aqueous $NaHCO_3$, dried, end evaporated under reduced pressure. The product was crystallized from *Et*OH (see Table 1 for yields).

Procedure for the bromination of aromatic amides
Compound **5** or **8** (3.1 mmol) was added to a stirred solution of 3.1 mmol of the aromatic amide in 15 cm^3 50% aqueous *DMF*. The mixture was stirred at rt for specified reaction time and then diluted with 100 cm^3 water. The precipitated product was filtered, washed with water, and crystallized (see Table 1 for yields). All bromo derivatives were additionally identified by ^{1}H and ^{13}C NMR.

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