The J-Like Structures of Azobenzene-Containing Amphiphiles, $C_{10}AzoC_5N^+Br^-$ and $C_8AzoC_5N^+Br^-$

Kenji Okuyama,* Chiyo Mizuguchi, Guiqing Xu, and Masatsugu Shimomura
Faculty of Technology, Tokyo University of Agriculture and Technology, Koganei, Tokyo 184

(Received March 20, 1989)

Totally synthetic azobenzene-containing amphiphiles $C_{10}AzoC_5N^+Br^-$ and $C_8AzoC_5N^+Br^-$ ($C_{31}H_{50}N_3O_3Br$ and $C_{29}H_{46}N_3O_3Br$ respectively) were crystallized from their solutions by the solvent-evaporation method. Both crystals are triclinic, with the space group of $P\bar{1}$ and $Z=2$. The unit cell dimensions of the former crystal are $a=3.233(5)$, $b=0.854(2)$, $c=0.608(1)$ nm, $\alpha=106.96(8)$, $\beta=86.6(1)$, and $\gamma=88.3(1)°$. Those of the latter crystal are $a=3.0098(6)$, $b=0.8553(2)$, $c=0.6084(1)$ nm, $\alpha=72.99(1)$, $\beta=92.57(2)$, and $\gamma=95.64(2)°$. The crystal structure of $C_{10}AzoC_5N^+Br^-$ was solved by the direct method and refined by the block-diagonal least-squares method: $R=0.082$ for 2927 observed reflections ($2\theta<120°$, Cu$K\alpha$, $\lambda=0.15418$ nm). On the other hand, that of $C_8AzoC_5N^+Br^-$ was refined by the full-matrix least-squares method: $R=0.058$ for 2993 reflections ($2\theta<120°$, Cu$K\alpha$). The molecular and crystal structures of these compounds are quite similar to those of the homologous compounds, $C_{12}AzoC_5N^+Br^-$ and $C_6AzoC_5N^+Br^-$. That is, the $C_nAzoC_5N^+Br^-$ molecules incline about 30° to the bilayer surface to pack in a tail-to-tail fashion. The azobenzene chromophore has a pronounced planarity which makes possible the parallel stacking of adjacent phenyl groups.

Totally synthetic azobenzene-containing amphiphiles with a single alkyl chain, $C_nAzoC_mN^+Br^-$ (Fig. 1), show several types of azobenzene chromophore-aggregation states, depending on the number of carbon atoms in the tail part (n) and the spacer part (m).[1] Different aggregation states of the azobenzene chromophores cause different types of orientation of the transition dipole of the long axis of the chromophore, detectable as a bathochromic or a hypsochromic shift of the absorption spectrum. According to a semiquantitative application of Kasha's molecular exciton theory,[2] the hypsochromic shift is attributable to the parallel (or side-by-side) orientation of the transition dipole (H-aggregate). This aggregation state was confirmed by an X-ray diffraction study of the cast films of $C_8AzoC_{10}N^+Br^-$.[3] On the other hand, the bathochromic shift is derived from the head-to-tail orientation between adjacent transition dipoles (J-aggregate). This type of aggregation was found in the single crystals, casted films, and the aqueous dispersion of $C_nAzoC_5N^+Br^-$ (n=12, 10, 8, and 6). In the cases of n=12 and 6, their crystal structures were determined in detail by the X-ray single crystal structure analyses and bore out the J-aggregation of azobenzene chromophores at the atomic level.[4,5]

In this paper, the crystal structures of the two compounds (n=10, 8) in the family of $C_nAzoC_5N^+Br^-$ will be determined for the further understanding of the stereochemical characteristics of the J-aggregation.

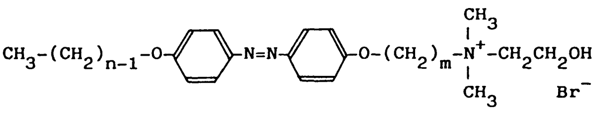

Fig. 1. Chemical formula of azobenzene-containing single chain amphiphiles (abbreviated as $C_nAzoC_5N^+Br^-$).

Experimental and Structure-Determination

Materials. The $C_{10}AzoC_5N^+Br^-$ (15 mg) was dissolved in a boiling solution (18 ml) of water and ethanol (water/ethanol=1/1 by volume ratio). Yellow, plate-like crystals with a thickness of about 0.02 mm were deposited from the solution by the solvent-evaporation method for about a week at room temperature. Single crystals of $C_8AzoC_5N^+Br^-$ were obtained by a similar method using a solution of ethanol and benzene (ethanol/benzene=1/2 by volume ratio).

Table 1. Crystal Data and Details of Experiments and Analyses

Compound	$C_{10}AzoC_5N^+Br^-$	$C_8AzoC_5N^+Br^-$
Formula weight	593.75	564.61
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a/nm	3.233(5)	3.0098(6)
b/nm	0.854(2)	0.8553(2)
c/nm	0.608(1)	0.6084(1)
α/°	106.96(8)	72.99(1)
β/°	86.6(1)	92.57(2)
γ/°	88.3(1)	95.64(2)
Cell volume/nm³	1.601(4)	1.4903(5)
Z	2	2
D_x/g cm^{-3}	1.232	1.263
D_m/g cm^{-3}	1.23	1.26
Radiation	Cu$K\alpha$	Cu$K\alpha$
μ(calcd)/cm^{-1}	17.83	18.50
Crystal size/mm³	0.5×0.4×0.01	0.5×0.2×0.01
Scan mode	ω	ω
Scan width/° (in ω)	3.4+0.15 tan θ	2.6+0.14 tan θ
Scan speed /° (in ω)min^{-1}	6	8
2θ range/°	2.5—120	2.5—120
No. of observed unique reflections	4498	3356
No. of reflections for R	2927	2993
R	0.082	0.058

X-Ray Work. The lattice parameters and diffraction intensities of $C_{10}AzoC_5N^+Br^-$ were measured on a Rigaku four-circle diffractometer with Ni-filtered Cu $K\alpha$ radiation (λ=0.15418 nm). Those of $C_8AzoC_5N^+Br^-$ were measured on the same type of diffractometer with graphite-monochromatized Cu $K\alpha$ radiation (RASA-5RII). The lattice parameters were refined by the least-squares fit using 31 reflections in the 2θ range of 10—50° for $C_{10}AzoC_5N^+Br^-$ and 25 reflections in the 2θ range of 45—50° for $C_8AzoC_5N^+Br^-$. Crystal data are listed in Table 1. Since the peak profiles of the reflections from the $C_{10}AzoC_5N^+Br^-$ crystal used for data collection were broken into two parts, a considerably wide scan-width was applied. The densities of the crystals were measured by the flotation method in a hexane and tetrachloromethane solution.

Three reference reflections, monitored every 50 reflections, showed no significant intensity-deterioration during the data collection of either sample. Corrections were made for the Lorentz and polarization factors, but not for the absorption and extinction effects. The experimental conditions are summarized in Table 1.

Structure-Determination. The structure of the $C_{10}AzoC_5N^+Br^-$ crystal was solved by the direct method with the MULTAN 78 program,[8] and the atomic parameters were refined by the block-diagonal least-squares method. On the other hand, the direct method with the SAPI 85 program[9] was applied to the structure of the $C_8AzoC_5N^+Br^-$ crystal and the atomic parameters were refined by the full-matrix least-squares method. The quantity minimized in the refinement was $\sum w(|F_o|-|F_c|)^2$, with w=1.0 in the case of $C_{10}AzoC_5N^+Br^-$ and with $w=1/(\sigma^2(F)+(0.01\times F_o)^2)$ in the other case. In the refinement, the reflections with $F_o<3\sigma$ were excluded from the reflection data. After several refinement cycles, the magnitudes of the observed structure factors of (h00) reflections were found to be consistently smaller than those of the calculated ones in both crystals. The same phenomenon was observed in the structure analyses of $2C_nN^+2C_1Br^-$ (n=18, 14);[6,7] it is attributable to the absorption effect originating from the very thin plate-like crystal shape and its long lattice constant normal to the plate

Table 2. Fractional Coordinates and Equivalent Isotropic Temperature Factors (B_{eq}) for Non-Hydrogen Atoms of $C_{10}AzoC_5N^+Br^-$ with Estimated Standard Deviations in Parentheses.

$B_{eq}=(4/3)\times\{\beta_{11}a^2+\beta_{22}b^2+\beta_{33}c^2+2(\beta_{12}ab+\beta_{13}ac+\beta_{23}bc)\}$

Atom	x	y	z	$B_{eq}\times 10^4$/nm^2
Br	0.92928(4)	0.3923(2)	0.0684(2)	503
O(1)	0.9971(2)	0.1883(8)	−0.3175(11)	464
O(2)	1.1965(2)	−0.4872(8)	−0.5756(11)	461
O(3)	1.3064(2)	−1.6236(8)	−0.0066(12)	530
N(1)	1.0678(2)	0.2073(9)	−0.7063(12)	369
N(2)	1.2383(2)	−1.0046(9)	−0.2324(13)	391
N(3)	1.2649(3)	−1.1077(10)	−0.3492(14)	433
C(1)	0.9928(3)	0.2268(12)	−0.5256(18)	442
C(2)	1.0296(3)	0.3143(10)	−0.5915(15)	366
C(3)	1.1010(4)	0.3204(15)	−0.7352(22)	608
C(4)	1.0589(4)	0.0955(15)	−0.9348(17)	580
C(5)	1.0829(3)	0.1094(11)	−0.5504(15)	343
C(6)	1.1209(3)	−0.0038(12)	−0.6480(16)	380
C(7)	1.1301(3)	−0.1102(12)	−0.4890(17)	409
C(8)	1.1622(3)	−0.2430(11)	−0.5979(16)	378
C(9)	1.1693(3)	−0.3523(11)	−0.4468(16)	389
C(10)	1.2057(3)	−0.6094(12)	−0.4796(16)	390
C(11)	1.1877(3)	−0.6219(12)	−0.2757(17)	435
C(12)	1.2000(3)	−0.7536(12)	−0.1977(17)	435
C(13)	1.2345(3)	−0.7290(12)	−0.6089(16)	398
C(14)	1.2463(3)	−0.8600(12)	−0.5359(16)	417
C(15)	1.2282(3)	−0.8741(11)	−0.3273(17)	387
C(16)	1.2744(3)	−1.2378(11)	−0.2550(16)	385
C(17)	1.2559(3)	−1.2517(11)	−0.0501(16)	379
C(18)	1.2677(3)	−1.3814(12)	0.0272(17)	448
C(19)	1.3034(3)	−1.3553(13)	−0.3793(17)	478
C(20)	1.3156(3)	−1.4876(12)	−0.3049(18)	480
C(21)	1.2970(3)	−1.5013(12)	−0.0997(17)	402
C(22)	1.3362(3)	−1.7533(13)	−0.1280(20)	535
C(23)	1.3392(3)	−1.8637(12)	0.0281(19)	498
C(24)	1.3715(4)	−2.0041(13)	−0.0584(20)	560
C(25)	1.3731(4)	−2.1084(14)	0.1037(22)	617
C(26)	1.4055(4)	−2.2454(14)	0.0377(23)	639
C(27)	1.4063(4)	−2.3512(16)	0.2011(24)	722
C(28)	1.4390(4)	−2.4852(17)	0.1460(27)	780
C(29)	1.4396(5)	−2.5871(18)	0.3123(27)	846
C(30)	1.4721(5)	−2.7232(21)	0.2491(32)	1054
C(31)	1.4729(7)	−2.8218(24)	0.4153(39)	1381

Table 3. Fractional Coordinates and Equivalent Isotropic Temperature Factors (B_{eq}) for Non-Hydrogen Atoms of $C_8AzoC_5N^+Br^-$ with Estimated Standard Deviations in Parentheses.

$B_{eq}=(4/3)\times\{\beta_{11}a^2+\beta_{22}b^2+\beta_{33}c^2+2(\beta_{12}ab+\beta_{13}ac+\beta_{23}bc)\}$

Atom	x	y	z	$B_{eq}\times 10^4$/nm^2
Br	1.07584(2)	−0.3539(1)	1.0478(1)	537
O(1)	1.0033(1)	−0.1856(5)	0.6800(7)	516
O(2)	0.7890(1)	0.3791(5)	0.4800(7)	520
O(3)	0.6718(2)	1.4502(5)	1.0845(7)	605
N(1)	0.9276(2)	−0.2435(7)	0.3137(10)	375
N(2)	0.7450(2)	0.8705(6)	0.8354(8)	469
N(3)	0.7157(2)	0.9593(6)	0.7265(9)	479
C(1)	1.0077(2)	−0.2229(8)	0.4711(12)	467
C(2)	0.9687(2)	−0.3280(9)	0.4156(12)	446
C(3)	0.8912(3)	−0.3852(12)	0.2988(20)	821
C(4)	0.9378(3)	−0.1283(10)	0.0802(12)	597
C(5)	0.9111(2)	−0.1546(8)	0.4717(11)	383
C(6)	0.8702(2)	−0.0643(9)	0.3875(11)	453
C(7)	0.8608(2)	0.0371(9)	0.5468(12)	438
C(8)	0.8255(2)	0.1544(8)	0.4460(11)	442
C(9)	0.8188(2)	0.2574(8)	0.6010(11)	430
C(10)	0.7793(2)	0.4955(8)	0.5766(11)	439
C(11)	0.7987(2)	0.5185(8)	0.7787(12)	463
C(12)	0.7860(2)	0.6418(9)	0.8576(12)	506
C(13)	0.7482(2)	0.6001(8)	0.4563(11)	474
C(14)	0.7360(2)	0.7227(8)	0.5350(11)	439
C(15)	0.7549(2)	0.7462(7)	0.7378(11)	409
C(16)	0.7060(2)	1.0836(7)	0.8240(11)	423
C(17)	0.7257(2)	1.1087(8)	1.0243(11)	465
C(18)	0.7131(2)	1.2301(8)	1.1059(12)	492
C(19)	0.6743(2)	1.1867(9)	0.7061(12)	519
C(20)	0.6616(2)	1.3109(9)	0.7867(12)	526
C(21)	0.6815(2)	1.3338(8)	0.9856(11)	474
C(22)	0.6402(3)	1.5627(9)	0.9750(13)	544
C(23)	0.6366(3)	1.6719(10)	1.1296(14)	587
C(24)	0.6019(3)	1.7927(11)	1.0487(15)	665
C(25)	0.5993(3)	1.8996(10)	1.2068(15)	683
C(26)	0.5646(3)	2.0193(11)	1.1373(17)	768
C(27)	0.5625(3)	2.1312(12)	1.2919(20)	769
C(28)	0.5258(3)	2.2421(13)	1.2343(19)	1040
C(29)	0.5212(5)	2.3496(16)	1.3803(28)	1364

surface and from the fairly large absorption coefficient. Therefore, the (h00) reflections with $h<9$ for C_{10}Azo-$C_5N^+Br^-$ and those with $h<10$ for $C_8AzoC_5N^+Br^-$ were excluded from the corresponding reflection data set at the final stage of the analyses. The final R values were 0.082 for all the non-hydrogen atoms and for the 46 hydrogen atoms of $C_{10}AzoC_5N^+Br^-$ and 0.058 for all the non-hydrogen atoms and for the 44 hydrogen atoms of $C_8AzoC_5N^+Br^-$. The final atomic parameters for non-hydrogen atoms are given in Tables 2 and 3.[10)]

The atomic scattering factors were taken from International Tables for X-Ray Crystallography, Vol. IV.[11)] Computations for $C_{10}AzoC_5N^+Br^-$ were done on an ACOS 700S computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University, and on a HITAC M-280H computer at the Computer Center, University of Tokyo, with the help of the UNICS III program.[12)] Those for $C_8AzoC_5N^+Br^-$ were done on an A-70 minicomputer with the aid of the CRYSTAN program in the RASA5R-II system (Rigaku Denki).

Results and Discussion

Molecular Conformation of $C_{10}AzoC_5N^+Br^-$ and $C_8AzoC_5N^+Br^-$. The bond distances and angles are shown in Fig. 2, together with their atomic labelling for non-hydrogen atoms. The numbering system of these atoms is the same as those of homologous compounds analyzed previously.[4,5)] The average C–C bond distance and C–C–C bond angle in the alkyl spacer and the tail parts are 0.151(3) nm and 113(1)° for $C_{10}AzoC_5N^+Br^-$, and 0.151(2) nm and 113(1)° for $C_8AzoC_5N^+Br^-$. These values are in good agreement with those reported previously for the long hydrocarbon chains of the amphiphilic compounds.[4–7,13,14)]

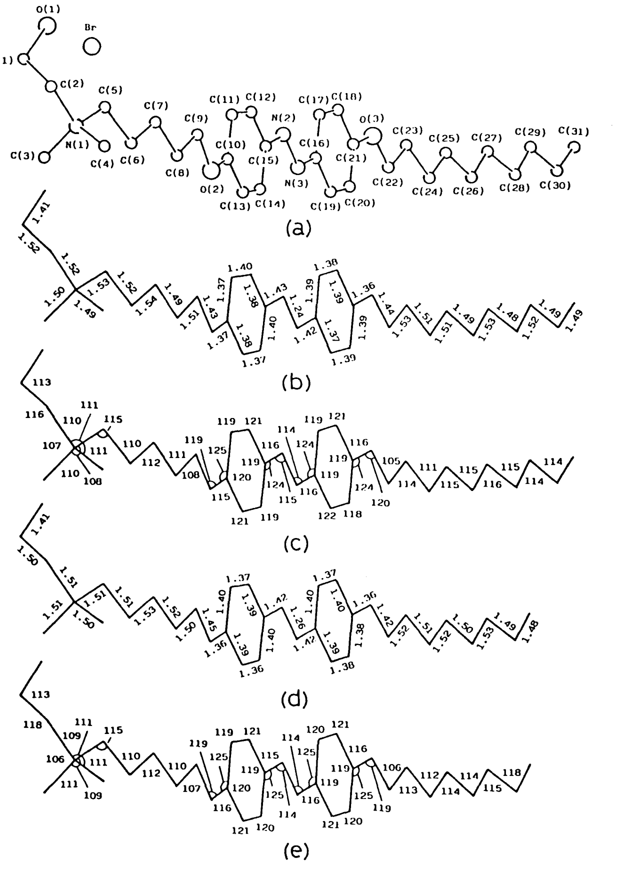

Fig. 2. Atomic labelling (a), bond lengths (1×10/nm) (b), and bond angles (/°) (c) in $C_{10}AzoC_5N^+Br^-$, together with those in $C_8AzoC_5N^+Br^-$ (d) and (e). Maximum estimated standard deviations for bond lengths and angles are 0.003 nm and 1° in $C_{10}AzoC_5N^+Br^-$ and 0.002 nm and 1° in $C_8AzoC_5N^+Br^-$.

The structural similarity of four homologous compounds, $C_nAzoC_5N^+Br^-$ (n=6, 8, 10, 12), clearly appears in the dihedral angles listed in Table 4. The dihedral angles of the four compounds are so similar to each other that the maximum deviation from the average is within 2° in every case. With the exception of the first two, all the dihedral angles have trans conformation. Especially, the planarity of the azobenzene moiety, which depends on the C(12)-C(15)-N(2)-N(3), C(15)-N(2)-N(3)-C(16) and N(2)-N(3)-C(16)-(19) angles, is very remarkable compared with those for other azobenzene-containing compounds, such as *trans*-azobenzene[15)] and 4,4′-dimethylazobenzene.[16)] The pronounced planarity of the azobenzene moiety must be one of the main features of the J-aggregation state of the $C_nAzoC_5N^+Br^-$ compounds. The alkyl tail part also makes a well-ordered zigzag plane. On the other hand, the zigzag plane in the alkyl spacer part is slightly distorted. This distortion may be ascribed to hydrogen bonds between terminal hydroxyl moieties and bromide anions, to electrostatic interactions between nitrogen cations and bromide anions, and to the existence of

Table 4. Dihedral Angles of $C_nAzoC_5N^+Br^-$

Dihedral angle/°	n=6	n=8	n=10	n=12	Average
O(1) C(1) C(2) N(1)	79	79	80	80	80
C(1) C(2) N(1) C(5)	−55	−56	−58	−57	−57
C(2) N(1) C(5) C(6)	180	179	180	179	180
N(1) C(5) C(6) C(7)	−174	−174	−173	−173	−174
C(5) C(6) C(7) C(8)	169	169	170	170	170
C(6) C(7) C(8) C(9)	−177	−177	−177	−178	−177
C(7) C(8) C(9) O(2)	173	173	173	172	173
C(8) C(9) O(2) C(10)	−176	−176	−176	−175	−176
C(9) O(2) C(10)C(13)	−174	−176	−176	−174	−175
C(12)C(15)N(2) N(3)	−178	−178	−177	−178	−178
C(15)N(2) N(3) C(16)	180	180	−179	180	180
N(2) N(3) C(16)C(19)	179	179	180	179	179
C(18)C(21)O(3) C(22)	180	180	179	180	180
C(21)O(3) C(22)C(23)	180	179	179	179	179
O(3) C(22)C(23)C(24)	−177	−176	−176	−176	−176
C(22)C(23)C(24)C(25)	−178	180	180	180	180
C(23)C(24)C(25)C(26)	−178	−178	−177	−177	−178
C(24)C(25)C(26)C(27)	180	−179	−179	−179	−179
C(25)C(26)C(27)C(28)		−176	−178	−177	−177
C(26)C(27)C(28)C(29)		179	179	179	179
C(27)C(28)C(29)C(30)			179	178	179
C(28)C(29)C(30)C(31)			179	−179	180
C(29)C(30)C(31)C(32)				180	180
C(30)C(31)C(32)C(33)				−179	−179

bulky methyl moieties.

Crystal Structure of $C_{10}AzoC_5N^+Br^-$ and $C_8AzoC_5N^+Br^-$. The packing arrangement viewed along the *c*-axis is shown in Fig. 3 ($C_{10}AzoC_5N^+Br^-$) and Fig. 4 ($C_8AzoC_5N^+Br^-$), together with the short interatomic distances between neighbouring chains and those between a bromide anion and the head portion of the amphiphile. The crystal structure of these two compounds can be seen as an alternation of the hydrophobic and hydrophilic layers stacked in the *a*-direction. Here, the hydrophilic and hydrophobic parts correspond to the bilayer surface and the inner part of the bilayer respectively.

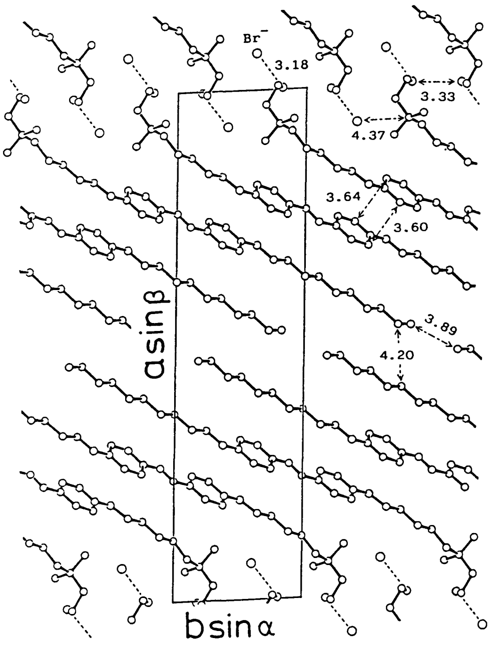

Fig. 3. Packing of $C_{10}AzoC_5N^+Br^-$ projected onto the *c*-plane (ORTEP[17]) drawing). Hydrogen bonds between bromide anions and hydroxyl groups are indicated by broken lines. Some short interatomic distances (1×10/nm) are shown.

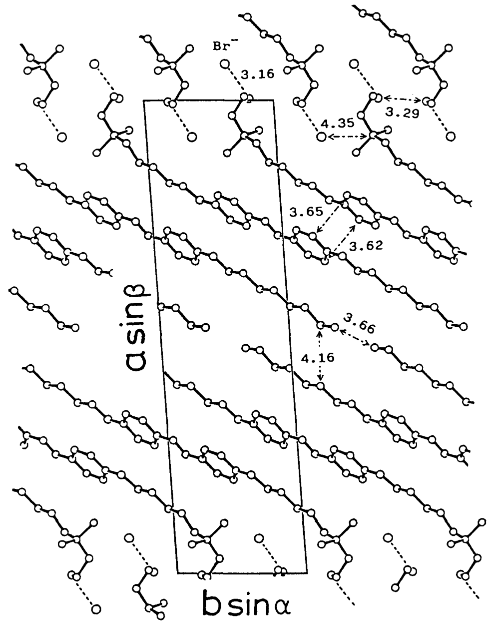

Fig. 4. Packing of $C_8AzoC_5N^+Br^-$ projected onto the *c*-plane.

In the hydrophobic layer, two molecules arranged in a tail-to-tail fashion and related by the inversion symmetry are inclined by about 30° to the layer surface. There is a remarkable tendency for the tail-to-tail distance to decrease along with the number of carbon atoms in the tail part of $C_nAzoC_5N^+Br^-$ compounds. That is, the tail-to-tail distances for $n=12$, 10, 8 and 6 are 0.391, 0.389, 0.366, and 0.356 nm respectively. This kind of systematic tendency was not found for the intermolecular distances in the other part of the amphiphile, such as the hydrogen-bond distances in the hydrophilic part and interatomic distances between adjacent azobenzene moieties.

In the hydrophilic layer, ammonium cations and bromide anions make a plane parallel to the bilayer surface. Two such planes are contained in one hydrophilic layer. The bromide anion is linked to the hydroxyl group of the adjacent molecule in a different bilayer by a hydrogen bond (0.318 nm in $C_{10}AzoC_5N^+Br^-$ and 0.316 nm in $C_8AzoC_5N^+Br^-$). Through this hydrogen bond, the head groups of amphiphiles in adjacent bilayers interdigitate mutually. In addition to the electrostatic interactions between the cationic ammonium nitrogens and the bromide anions, this interdigitated structure in the hydrophilic layer must contribute to the stabilization of crystals. On the other hand, the hydrophobic alkyl chains and azobenzene moieties are stabilized only by the van der Waals interactions. Therefore, the above tendency for the tail-to-tail distance in the hydrophobic part to decrease is attributable to the increase in the influence of various strong interactions in the hydrophilic part as shortening the tail length in $C_nAzoC_5N^+Br^-$.

The authors wish to express their deep thanks to Professor Yukiteru Katsube at the Crystallographic Research Center, Institute for Protein Research, Osaka University, for the use of the Institute's facilities to analyse one of the specimens.

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