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Difference in micellar properties of sodium dodecyl sulfonate from sodium 4-decyl naphthalene sulfonate in D_2O solution studied by ^1H NMR relaxation and 2D NOESY

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Abstract ^1H chemical shift changes of sodium 4-decyl naphthalene sulfonate (SDNS) at 313 K show that its critical micellar concentration lies between 0.82 and 0.92 mmol/dm^3, which is in the same range as that of the previous study at 298 K. The spin–lattice relaxation time, spin–spin relaxation time and two-dimensional nuclear Overhauser enhancement spectroscopy experiments give information about the structure of the SDNS micelle and the dynamics of the molecules in the micelle. The size of the SDNS micelle remains almost unchanged in the temperature range from 298 to 313 K as deduced by analyzing the self-diffusion coefficient. Special arrangement of the naphthyl rings of SDNS in the micelles affects the packing of these hydrophobic chains. The methylene groups of the alkyl chain nearest the naphthalene groups penetrate into the aromatic region, which results in a more tightly packed hydrophobic micellar core than that of sodium dodecyl sulfonate.

Keywords Surfactants · Micelle · Sodium dodecyl sulfonate · Sodium 4-decyl naphthalene sulfonate · ^1H NMR relaxation

Introduction

Both sodium dodecyl sulfonate (SDSN) and sodium 4-decyl naphthalene sulfonate (SDNS) are anionic surfactants and SDSN has been widely used in many application fields, such as detergency, painting, coatings, cosmetics, biochemical solvents, and oil recovery. Micellar chemical flooding using surfactants is a necessary development in enhanced recovery of oil [1, 2]. On one hand, the surfactant produces ultralow surface tension between oil and water; on the other hand, the ability of the surfactant to solubilize various organic compounds which are sparingly soluble or insoluble in water can be utilized to decrease the dose of surfactants [3, 4, 5]. Most of the previous investigations focused on the characteristics of straight-chain alkyl sulfonates or the effects of headgroups of surfactants on the micellar framework above their critical micellar concentrations (cmc) [6, 7, 8]. The SDNS surfactant has not been studied enough in comparison with SDSN. SDNS is different in structure from SDSN. The polar group of SDNS is adjacent to a naphthyl ring. This instead of a methylene group can affect greatly the property of the palisade layer. Investigations by many kinds of experimental methods, such as small-angle X-ray scattering, small-angle neutron scattering, and 1D NMR [9, 10, 11, 12], show that the micelle is composed of a hydrophobic core region, a palisade layer, and a Stern layer. In addition, the core region is composed of a liquidlike hydrocarbon into which water does not penetrate deeply [7, 8, 13, 14]. These properties also demonstrate that the surface region of a roughly spherical micelle is not completely covered by polar headgroups and there exist contacts of the alkyl chain of a surfactant with water molecules on the surface of the hydrophobic core. The microstructure of the palisade layer, which is important for the formation and the characteristic of micelles, has been studied extensively [15, 16]. NMR spectroscopy is a versatile technique for the study of surfactants [17]. It provides information at a molecular level that is not available by other spectroscopic methods. Recently we have studied the behavior of SDNS in aqueous solution by 1D and 2D NMR at 298 K in detail, and provided information about the relative arrangement of the naphthyl rings in the micelles [18]. In this work we intended to acquire information about the influence of temperature on the properties of SDNS and the difference between SDNS and SDSN in their properties including the micellar structures.

Experimental

SDSN was of chemically pure grade from the Yongjia Fine Chemical Plant of China. SDNS was synthesized by the Technical Institute of Physics and Chemistry. D_2O (99.8% deuterated) was a product of Wuhan BoPu Corporation of the Chinese Acedemy of Sciences. NMR measurements were performed using a UNITY INOVA-600 spectrometer with a ^1H frequency of 599.904 MHz. D_2O was used as a solvent instead of water in order to weaken the water signal, and the presaturation method was used to further suppress the ^1H signal of the solvent. A Double-pulse sequence was used to measure the chemical shifts of protons, and an inversion recovery method and Carr–Purcell–Meiboom–Gill pulse sequences were used for the spin–lattice relaxation time, T_1, and spin–spin relaxation time, T_2, measurements, respectively. 2D NOESY experiments were performed with the standard three-pulse sequence [19] with mixing times of 700 ms for SDSN solution at a concentration of 5 times its cmc and with different mixing times of 500, 700, and 1000 ms for SDNS solution at a concentration of 2.59 mmol/dm^3, respectively.

Results and discussion

Physicochemical properties of SDNS

In order to compare the micellar properties of SDNS with those of SDSN ^1H NMR parameters were measured at 313 K, above the Krafft point of SDSN, 310 K.

Dependence of the cmc of SDNS on temperature

Similarly to Ref. [18], ten resolvable resonance peaks were resolved in the SDNS ^1H NMR spectrum and the assignment of these peaks follows Ref. [18]. They are a schematic representation of ten kinds of chemically similar groups of spins. H1–H10 stand for the corresponding proton signals as indicated by the formula shown in Fig. 1.

It is obvious from Fig. 2 that the chemical shifts of the some of the SDNS protons changed dramatically in the concentration range between 0.82 and 0.92 mmol/dm^3. This implies that there are changes in the chemical environments of the SDNS protons. Therefore, the cmc of SDNS at 313 K, which lies between 0.82 and 0.92 mmol/dm^3, is in the same range as that at 298 K [18], as shown in Table 1 and Fig. 2.

Dynamics of SDNS protons at 313 K

T_1, T_2, and T_R (T_2/T_1) of ^1H NMR of SDNS in aqueous solution at a concentration of 2.59 mmol/dm^3 (3 times its cmc) are listed in Table 2. That the T_R values of the hydrophobic protons are far less than unity suggests that the motion of the protons becomes more restricted and that the SDNS molecules have formed micellar aggregates [20]. For example, H7, one of these protons of the straight-alkyl chain, whose T_R values is only 0.07, is a typical proton involved in the palisade layer [20, 21]. We can also notice that H2, H4,5, and H6 posses lower values of T_R than those of the protons of H1 and H3. This implies that the protons of H2, H4,5, and H6 are located more compactly. The T_R value of the H8

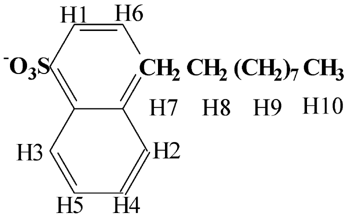


Fig. 1 Formula of sodium 4-decyl naphthalene sulfonate (*SDNS*) with proton numbering

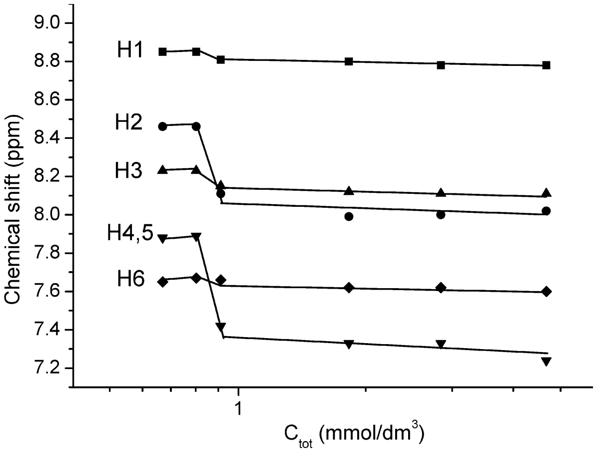


Fig. 2 The chemical-shift dependence of SDNS protons on the concentration at 313 K

Table 1 The variation of the chemical shift (ppm) of sodium 4-decyl naphthalene sulfonate (*SDNS*) protons at 313 K with concentration in aqueous solution

[SDNS] (mmol/dm^3)	H1	H2	H3	H4,5	H6	H7	H8	H9	H10
0.70	8.85	8.46	8.23	7.88	7.67	3.33	1.92	1.41	1.01
0.82	8.85	8.46	8.23	7.89	7.67	3.33	1.93	1.42	1.02
0.92	8.81	8.09	8.15	7.42	7.71	3.11	1.76	1.39	1.02
1.68	8.80	7.99	8.12	7.33	7.62	2.97	1.67	1.37	1.02
2.59	8.78	7.98	8.11	7.31	7.62	2.96	1.66	1.36	1.01
4.16	8.78	7.96	8.11	7.24	7.60	2.89	1.64	1.36	1.02

Table 2 T_1, T_2, and T_R (T_2/T_1) of 2.59 mmol/dm^3 SDNS solution at a constant temperature of 313 K. The experimental error is less than 2%

	H1	H2	H3	H4,5	H6	H7	H8	H9	H10
T_1 (ms)	546.2	793.1	556.4	806.8	793.8	449.7	525.7	793.9	1393
T_2 (ms)	89.19	30.75	59.06	32.99	45.88	31.12	58.7	153.3	367.9
T_R	0.16	0.04	0.11	0.04	0.06	0.07	0.11	0.19	0.26

protons shows that their motion is less restricted than the protons of H7.

In our previous study, simulation of the decay curves of proton relaxation for micellar solutions showed that the relaxation decay curves of T_1 are exponential, while the curves of T_2 of some of the protons on the hydrophobic chains are biexponential. The fast relaxing component is attributed to the protons situated near or on the surface of the micellar core, while the slower one is attributed to the remaining protons in the interior [16]. The simulation results of H1, H2, H3, H4,5, H6, H7, and H8 are listed in Table 3. The simulation of the relaxation decay of H8 protons manifests biexponential behavior and one of the T_2 values is of the order of several tens of microseconds, the same as the T_{2f} values of the protons H2, H4,5, H6, which form the palisade layer. This shows that some of the H8 protons participate in the formation of the palisade layer and also that the protons of H8 reside in two different sites and transfer between both sites in mobile equilibrium on any occasion. It is well known that the hydrophobic effect expels the hydrocarbon chains of the surfactants from the aqueous medium to avoid hydrocarbon–water contact, but the naphthyl rings show steric hindrance, which causes the palisade layer to be less tightly packed [22, 23]. So the protons of H7 and H8 should be embedded in the aromatic ring environment to reduce the water penetration into the hydrophobic core. However, Table 3 shows a great difference between the protons of H7 and H8. It suggests that all of the protons of H7 take part in the formation of the palisade layer, whereas only 62% of H8 protons exist in this layer. Besides the majority of the protons of H2, H4,5, and H6 and a fraction of the protons of H1 and H3 participate in the formation of the palisade layer, as shown in Table 3.

Table 3 Behavior of the T_2 relaxation decay in the SDNS solution at a concentration of 3 times its critical micellar concentration (*cmc*) at 313 K. T_{2f} and T_{2s} stand for the T_2 value of the fast relaxing component and that of the slow relaxing part, respectively, and P_{2f} stands for the percentage of the protons exhibiting fast relaxation compared to the total number of protons

	H1	H2	H3	H4,5	H6	H7	H8
T_{2f} (ms)	37.85	26.91	31.39	31.13	34.38	31.15	42.81
T_{2s} (ms)	96.22	265.25	64.76	191.23	84.67	–	92.73
P_{2f} (%)	10	92	20	96	70	100	61

Influence of temperature on the T_2 relaxation times of different protons

It is well known that the increase in temperature enhances the Brownian movement of the molecules in the solution and increases the T_2 values of their protons. We measured the T_2 values at 298, 303, and 313 K as listed in Table 4. It is obvious that the T_2 of H1, H3, H9, and H10 got longer as the temperature increased; however, the reverse has been observed for H2, H4,5, H6, H7, and H8, which are those protons that participate in the formation of the palisade layer shown in Ref. [18]. This implies that in order to prevent the enhanced penetration of water by increased temperature the protons situated on the surface layer of the hydrophobic micellar core become more tightly packed.

Table 4 T_2 values (ms) of SDNS protons at different temperatures of 298, 303, and 313 K. The experimental error is less than 2%

T (K)	H1	H2	H3	H4,5	H6	H7	H8	H9	H10
298	64.33	48.04	45.8	43.19	56.62	34.1	54.5	105.1	245.3
303	72.27	42.42	50.33	40.64	53.79	35.17	59.67	120	277.2
313	89.19	30.75	59.06	32.99	45.88	31.12	58.7	155.3	367.9

2D NOESY experiment of SDNS

In order to examine the evolution of cross-peaks in the 2D NOESY experiments of SDNS at a concentration of 3 times its cmc, different mixing times of 500, 700, and 1,000 ms were chosen. Their contour plots are shown in Fig. 3a–c. Following Fig. 3, the cross-peaks between protons on adjacent carbon atoms (H1–H6, H2–H3, etc.) were observed. The intensities of the cross-peaks between H4 and H6 and between H3 and H5 for different mixing times of 500 ms, 700 ms, and 1 s are 20.58, 22.94 and 23.17, and 4.37, 6.78, and 6.32, respectively. The intensity of the cross-peak of H3–H5 reached its maximum at a mixing time of 700 ms, and probably it has already decreased at this time. So it is reasonable to choose a mixing time of 500 ms to estimate the distances to ensure that the intensities of the cross-peaks are not destroyed by spin diffusion and remain high enough to get fine definition. One can extract valuable information about interproton distances from the intensities of the cross-peaks with the aid of one known distance in the molecule [21]. The following formula was used with the aid of the known distance H1–H6 in the molecule and the corresponding cross-peak signal intensity:

$$\sqrt[6]{\frac{I_{16}}{I_{mn}}} = \frac{r_{mn}}{r_{16}}, \qquad (1)$$

where, I_{16} and I_{mn} are the intensities of the cross-peaks between H1 and H6 and between the two nuclei of interest, respectively; r_{16} and r_{mn} are the corresponding interproton distances. The chemical shifts of the methylene protons of $(CH_2)_7$, H9, overlap greatly, and the intensities of the cross-peaks of H9 with other protons are due to contributions from the sum of the seven methylene segments and are not suitable to estimate the interproton distances. The interproton distances in the micellar state calculated using Eq. (1) and those using HYPERCHEM on the basis of a single molecular state are shown in Table 5.

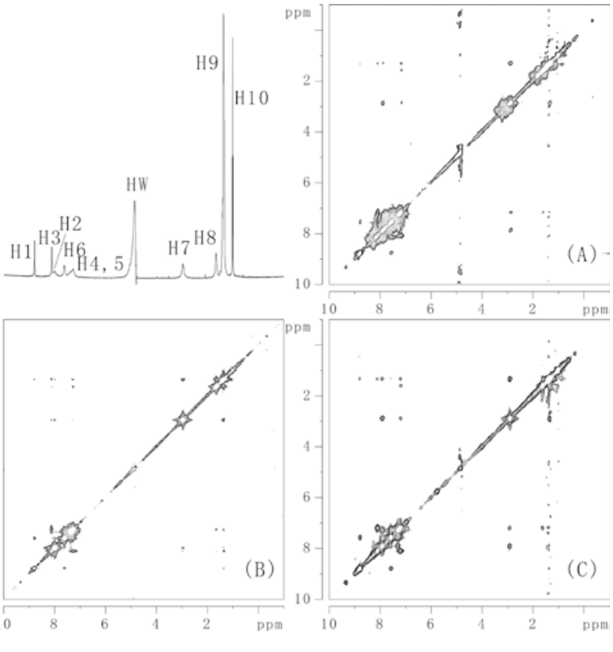


Fig. 3 Contour plots of 2D NOESY spectra of 2.59 mmol/dm^3 SDNS solution at 313 K with mixing times of **a** 500 ms, **b** 700 ms, and **c** 1 s

The relative arrangement of the molecules of SDNS in the micelles at 298 K was described in a previous study [18]. The unsubstituted halves of the naphthyl rings are embedded between the halves substituted by the sulfonate groups of other SDNS molecules with the naphthyl rings packed radiating outwards. Following Table 5, it is obvious that the interproton distances between H1–H6, H3–H5, H2–H4, and H7-H8, which are on adjacent carbon atoms, remain almost unchanged upon micellization within 10% error at different temperatures compared with those in the single molecular state. It also shows the significant decrease in interproton distances between H6 and H2 and between H6 and H4 at 313 K compared to the distances observed at 298 K. Moreover, it is obvious from Fig. 3 that there are cross-peaks between the H9 protons and the H3 and H4,5 protons, respectively; these show that the interproton distances are less than 5 Å. All these facts suggest that the naphthyl ring arrangement in the SDNS micelle at 313 K agrees with that at 298 K; however, comparing the interproton distances of SDNS at 313 K with those at 298 K, one observes that the distances between H4 and H7 and between H4 and H8 are obviously shortened. This implies that the alkyl chains of SDNS penetrate more tightly into the aromatic region at 313 K than at 298 K. The decrease in interproton distances should originate from the enhanced hydrophobic effect, which increases along with the penetrability of solvent as the temperature increases, to avoid hydrocarbon–water contact.

Table 5 Interproton distances (Å) of SDNS micelles at a concentration of 3 times the cmc calculated from the cross-peak intensities of the 2D NOESY spectrum at a mixing time of 500 ms and by HYPERCHEM at 313 K. The interproton distances were calculated by assuming the distance between H1 and H6 is 2.4 Å, taken from the value calculated by HYPERCHEM, and r_{HYP} represents the interproton distance calculated by HYPERCHEM on the basis of a single molecular state

	r_{mn}^{298} (Å)	r_{mn}^{313} (Å)	r_{HYP} (Å)		r_{mn}^{298} (Å)	r_{mn}^{313} (Å)	r_{HYP} (Å)
H1–H6	2.4	2.4	2.40	H7–H8	–	2.6	2.80
H2–H4	2.6	2.5	2.44	H3–H8	–	3.9	7.39
H2–H6	2.4	2.3	4.84	H4–H6	2.0	1.8	7.00
H2–H7	–	2.3	2.32	H4–H7	3.5	2.5	6.18
H3–H5	2.2	2.0	2.43	H4–H8	3.7	3.2	6.95

Self-diffusion coefficient of SDNS

Using self-diffusion measurements, one can compare the sizes of the roughly spherical micelles. Self-diffusion coefficients of SDNS protons, D_{obs}, at 3 times the cmc and at the single molecular state (0.82 mmol/dm^3) at 298, 303, and 313 K are listed in Table 6. It should be emphasized that these values are averages of the self-diffusion coefficients of all the protons in the molecule.

Amphiphilic chemical exchange of molecules between the monomeric and micellar states is rapid on the NMR time scale in aqueous solution, so the phase-separation model of micelle formation, which is based on the assumption that above the cmc the monomer concentration is nearly constant, can be used to interpret the phenomenon with the following formula [14, 24]:

$$D_{\mathrm{obs}}=pD_{\mathrm{mon}}+(1-p)D_{\mathrm{mic}}, \tag{2}$$

where D_{obs}, D_{mon}, and D_{mic}, respectively, stand for the self-diffusion coefficients of the observed value, the monomeric state, and the micellar state of the same proton groups, and p is the ratio of the concentration of monomeric states to the total molecule concentration of the SDNS solution at a concentration above its cmc. The average of the self-diffusion coefficient at 3 times its cmc (D_{obs}) and that at the single molecular state at a concentration of 0.82 mmol/dm^3 (D_{mon}) are used in this formula. The change of the viscosity of the medium is very small when the temperature varies for the SDNS dilute solution, i.e. the viscosity is nearly a fixed value. Fig. 4 shows that the temperature dependence of D_{mic} is an approximate straight line, which implies that the increase of D_{mic} with temperature obeys the Stokes–Einstein formula. Consequently the size of the micelle remains almost unchanged in the temperature range from 298 to 313 K.

Table 6 Variation in the self-diffusion coefficient (10^{-10} m^2/s) of SDNS at a concentration of 3 times its cmc with temperature

T (K)	D_{obs}	D_{mon}	D_{mic}
298	1.3421	3.5243	0.3765
303	1.705	4.2372	0.6584
313	2.4341	5.8828	1.0647

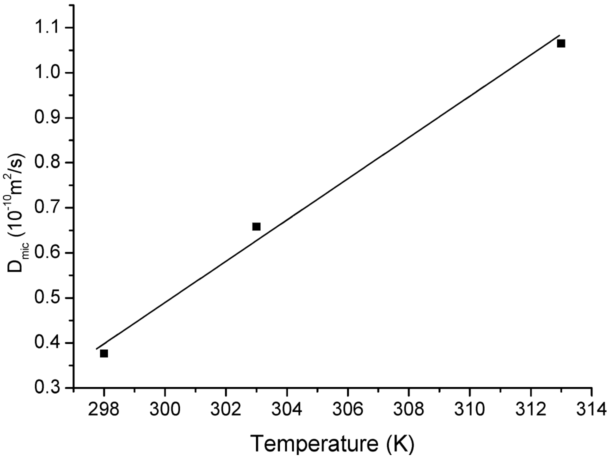
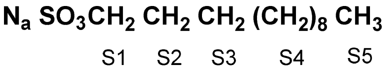


Fig. 4 The beeline of the calculated self-diffusion coefficient of SDNS micelles, D_{mic}, versus temperature at a concentration of 3 times its critical micellar concentration (*cmc*) at 313 K

^{1}H NMR relaxation of SDSN and 2D NOESY experiment

There are five resolvable resonance peaks, corresponding to five kinds of hydrocarbon groups with similar chemical shift, respectively, in the SDSN ^{1}H NMR spectrum. S1–S5 stand for the corresponding proton signals as indicated by the formula shown in Fig. 5.

T_1 and T_2, and T_R of SDSN protons in D$_2$O solution at a concentration of 5 times the cmc are listed in Table 7. T_R of all the proton signals of SDSN is about 0.2 at 313 K. It suggests that the motion of SDSN protons is restricted and that these protons are closely packed in the micellar core [10]. The sulfonate groups take part in the formation of the palisade layer [7]; thus one observes that the T_2 value of S1 protons is the smallest among the five signals and that the T_2 values of the hydrophobic protons increase as their distances to the polar groups get longer [11]. Simulation of S1, S2, and S3 spin–spin relaxation decay curves shows that the exponential simulation can fit the T_2 relaxation decay nicely. One can observe that the T_R values of SDSN are higher than those of SDNS at 313 K. This suggests that the motion of the SDSN protons is freer than that of the SDNS protons. The reason for this originates from the difference in the chemical structures of the surfactants. In forming micelles, owing to the existence of the naphthyl ring, adjacent to the SO$_3^-$ group, the area occupied by each polar head of SDNS on the surface



$$\mathrm{N_a\,SO_3\underset{S1}{CH_2}\,\underset{S2}{CH_2}\,\underset{S3}{CH_2}\,\underset{S4}{(CH_2)_8}\,\underset{S5}{CH_3}}$$

Fig. 5 Formula of sodium dodecyl sulfonate (*SDSN*) with proton numbering

Table 7 T_1, T_2, andT_R of sodium dodecyl sulfonate (*SDSN*) protons at a concentration of 5 times the cmc at 313 K. The experimental error is less than 2%

	S1	S2	S3	S4	S5
T_1 (ms)	1,173	1,040	886.8	1,014	1,720
T_2 (ms)	206.5	224.9	236.5	233.7	285
T_R	0.17	0.22	0.27	0.23	0.16

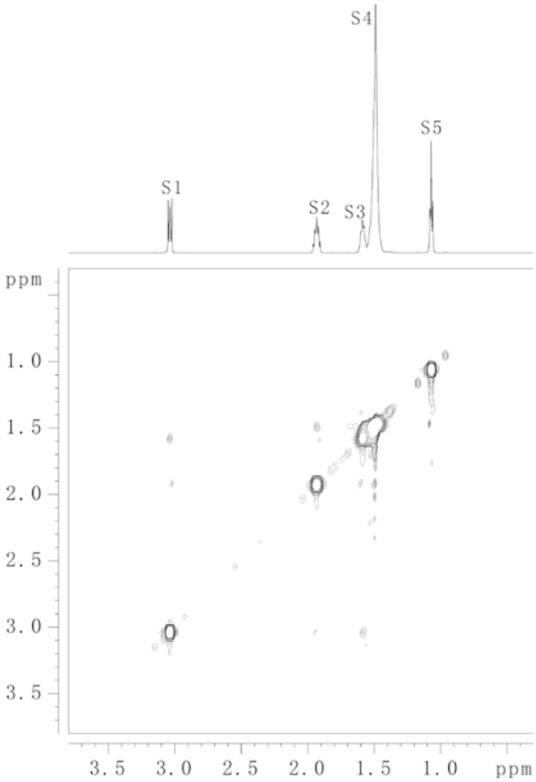


Fig. 6 Contour plot of the 2D NOESY map of SDSN solution at a concentration of 5 times its cmc at 313 K with a mixing time of 700 ms

layer of the hydrophobic micellar core is larger than that of SDSN, so the repulsion between the negatively charged sulfonate ions is weakened by the increased distance between the polar heads in SDNS micelles compared to in SDSN micelles. Consequently, the density of the protons in the palisade layer of the SDSN micelle is smaller and the SDSN micelle begins to appear in a higher concentration in aqueous solution than the SDNS micelles.

Table 8 Interproton distances (Å) of SDSN micelles at a concentration of 5 times the cmc calculated from the cross-peak intensities of the 2D NOESY spectrum at a mixing time of 700 ms and by HYPERCHEM at 313 K. The interproton distances were calculated by assuming the distance between S1 and S2 is 2.76 Å, taken from the value calculated by HYPERCHEM, and r_{HYP} represents the interproton distance calculated by HYPERCHEM on the basis of a single molecular state

	S1–S2	S1–S3	S2–S3
r_{mn}	2.76	2.30	2.56
r_{HYP}	2.76	2.80	2.77

The 2D NOESY spectrum for SDSN aqueous solution obtained with a mixing time of 700 ms is shown in Fig. 6. Cross-peaks between protons on adjacent carbon atoms (S1–S2, S2–S3, etc.) are observed. r_{mn} is calculated from the corresponding intensities of the cross-peaks with the aid of the known S1–S2 distance in the molecule, and r_{HYP} is obtained in the previously mentioned way. The values are shown in Table 8. Comparing r_{mn} with r_{HYP}, it is obvious that the interproton distances of S1–S2 and S2–S3 remain almost unchanged upon micellization within 10% error. However, the interproton distance of S1–S3 is shortened from Table 8; this suggests that the alkyl chain is twisted in the micellar state.

Summary

We have shown that the cmc of SDNS, the arrangement of naphthyl rings in the palisade layer is the same as at 298 K, and the size of the SDNS micelle are almost unchanged at 313 K. But it is obvious that the alkyl chains of SDNS are snaky and the first and second methylene groups next to the naphthalene rings are packed more tightly among the naphthyl rings in the palisade layer at 313 K. The alkyl chain of SDSN is twisted in the micellar state, but the palisade layer of SDSN is packed more loosely than SDNS at 313 K.

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