Synthesis and Characterization of Cleavable Cationic Surfactants with a 1,3-Dioxane Ring

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ABSTRACT: A series of cationic cleavable surfactants was prepared by condensation of 2-alkyl-1,3-propanediols with 3-bromopropanal, followed by reaction with triethylamine. Each surfactant is a mixture of two diastereomers, and their precursors' stereochemical assignments were based on ^{1}H nuclear magnetic resonance spectra and melting points. The critical micelle concentrations, Krafft points and destructibility of these surfactants were determined.

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KEY WORDS: Critical micelle concentration, 1,3-dioxane ring, Krafft point, quaternary ammonium cleavable surfactants, stereochemistry.

Cleavable surfactants were designed so as to avoid formation of troublesome emulsions of conventional surfactants and to facilitate extraction of desired products during reaction mixture work-up. In 1980, Cuomo *et al.* (1) reported the destruction of disulfide surfactants with dithioerythritol. Since then, many surfactant chemists have devoted their efforts to prepare and characterize various new types of cleavable surfactants. In particular, the Jaeger group (2–8) and Yamamura group (9–14) have paid much attention to cleavable surfactants with a 1,3-dioxolane ring. By contrast, only a few publications have dealt with cleavable surfactants with a 1,3-dioxolane ring (15–17). To compare with surfactants bearing a 1,3-dioxolane ring, our interest in cleavable surfactants has focused on those with a 1,3-dioxane ring, and we have recently reported two series of dianionic cleavable surfactants of such structure (18,19). All of the reported cleavable surfactants with a 1,3-dioxane ring are anionic or nonionic surfactants, and, to our knowledge, no cationic surfactants with a 1,3-dioxane ring have appeared. In this report, we present the preparation and properties of the first class of cationic surfactants with a 1,3-dioxane ring.

EXPERIMENTAL PROCEDURES

Materials. 2-Alkyl-1,3-propanediols (**1**) were synthesized by reaction of alkyl bromides with ethyl malonate in the presence of excess anhydrous K_2CO_3 and a catalytic amount (2.5 mol%) of Bu_4NBr, followed by reduction with $LiAlH_4$ (20). The chemical structure of diols **1** was confirmed by ^{1}H nuclear magnetic resonance (^{1}H NMR; 400 MHz, $CDCl_3$ as solvent with Me_4Si as internal standard) and infrared (IR) spectroscopy. 3-Bromopropanal was prepared *in situ* before use (21). All other chemicals were of analytical grade, and triethylamine and acrolein were distilled before use.

Synthesis of 5-alkyl-(2-bromoethyl)-1,3-dioxanes (**3a–d**). 5-Undecyl-(2-bromoethyl)-1,3-dioxane (**3a**) was synthesized as follows: A mixture of 5.3g (95 mmol) acrolein, 150 mL CH_2Cl_2 and 0.1 g dicinnamalacetone as indicator was cooled to 0–5°C while being magnetically stirred under nitrogen atmosphere. Dried hydrogen bromide was bubbled into the yellow solution until the color turned into deep red, which persisted for at least 5 min after the gas had been turned off (21). Then 21.0 g (86 mmol) 2-dodecyl-1,3-propanediol and 0.4 g *p*-toluenesulfonic acid monohydrate (TsOH) were added. The mixture was stirred at ambient temperature for 24 h. CH_2Cl_2 was distilled off, and the residue was dissolved in 75 mL diethyl ether, washed with two 50-mL portions of saturated aqueous $KHCO_3$ and dried over anhydrous $K_2CO_3$for 4 h. After filtration, the filtrate was evaporated to give 28.8 g crude product. Of this, 1.91 g was separated on a 2 × 60 cm column of silica gel (120–180 mesh) with 3 vol% diethyl ether in petroleum ether (60–90°C) as eluent to give 0.56 g of **3a** *(cis)* ($R_f = 0.27$), 0.63 g of **3a** (*trans*) ($R_f = 0.22$), 0.34 g of a mixture of **3a** (*cis*) and **3a** (*trans*) and some by-products. The eluent used for thin-layer chromatography (TLC) was the same as that for column chromatography. Each diastereomer was further purified by recrystallization from acetone to give **3a** (*trans*), melting point (m.p.) 40–41°C and **3a** (*cis*), m.p. 29–30°C. **3b–d** were prepared by the same procedure except for distillation under reduced pressure instead of column chromatography.

Synthesis of quaternary ammonium surfactants **4a–d**. **4a** (trans) was prepared by the following procedure: A mixture of 1.80 g of **3a** (*trans*) and 10 mL triethylamine were re-

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fluxed with stirring for 5 d. After cooling, the resultant precipitate was filtered with suction, washed with diethyl ether, affording 1.44 g of white solid **4a** (*trans*) (63%), which was further purified by recrystallization from a mixture of ethanol and ethyl acetate. Other surfactants were prepared by the same procedure.

Determination of critical micelle concentration (CMC) of surfactants **4a–d**. The CMC was determined by the sharp break on the I_3/I_1 of pyrene vs. surfactant concentration curve (22).

Krafft points of surfactants **4a–d**. Krafft points were measured as the temperature at which a 1% dispersion becomes clear on gradual heating (23).

Destructibility of surfactants **4a–d**. A mixture of a weighed amount of surfactant and 25 mL of 2 N HCl was magnetically stirred at 50°C for a given time. After cooling, the reaction mixture was extracted with three 25-mL portions of diethyl ether, and the combined extracts were dried over anhydrous Na_2SO_4 for 3 h. Then, Na_2SO_4 was filtered off, and the filtrate was evaporated to give the corresponding 2-alkyl-1,3-propanediol.

RESULTS AND DISCUSSION

A series of quaternary ammonium-type cleavable surfactants **4a–d** were simply prepared by condensation of 2-alkyl-1, 3-propanediols with 3-bromopropanal, followed by reaction with triethylamine (Scheme 1).

Yamamura *et al.* (9) have previously reported the synthesis and properties of 2-methyl-2-alkyl-4-triethylammoniomethyl-1,3-dioxolane iodides, which were prepared by reaction of 2-methyl-2-alkyl-4-bromomethyl-1, 3-dioxolanes with diethylamine and subsequent quaternization with ethyl iodide. In Jaeger's group, the attempt to react a chloro ketal with triethylamine or triethylamine/ethanol to incorporate a quaternary ammonium head group failed (2). However, we successfully prepared a series of quaternary ammonium surfactants, obtained directly from the corresponding bromides in refluxing triethylamine.

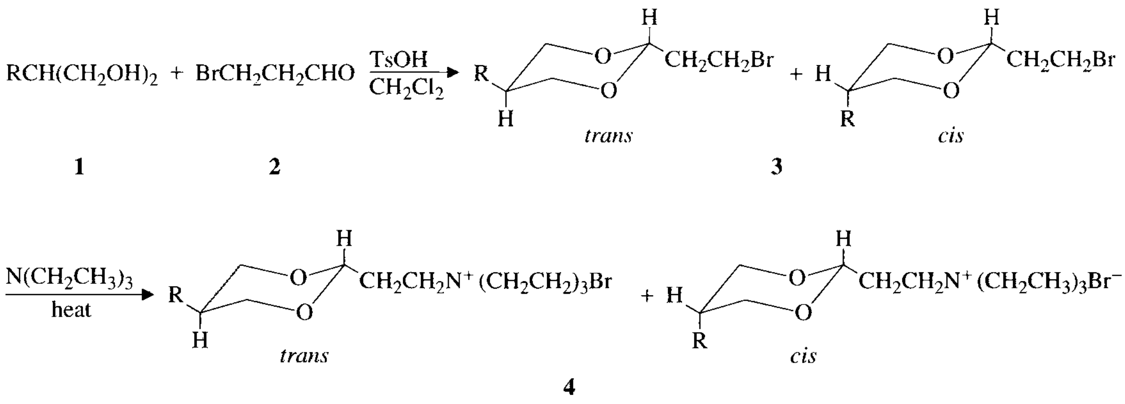
Both **3a–d** and **4a–d** were a mixture of two diastereomers. The yields and *trans*-to-*cis* ratios of bromides **3a–d** are listed in Table 1. The yields, melting points, CMCs and *trans*-to-*cis* ratios of surfactants **4a** (*trans*), **4a** (*cis*) and **4a–d** are shown in Table 2.

All of bromides **3a** (*trans*), **3a** (*cis*), **3b–d** and surfactants **4a** (*trans*), **4a** (*cis*), **4a–d** were characterized by ^1H NMR, IR spectroscopy and elemental analysis (Tables 3 and 4).

The stereochemical assignments of two diastereomers of bromides **3a–d** were based on their ^1H NMR spectra, TLC characteristics and melting points. For **3a** (*trans*), the axial hydrogens (H_a) next to oxygens of the 1,3-dioxane ring were split by the germinal equatorial hydrogen (H_e) and the H_a on the adjacent carbon; these two H_as appeared as a triplet because of the same coupling constant. The corresponding H_es absorbed at even lower field, coupled with the germinal H_a and adjacent H_a. The J value of the former was greater than that of the latter. Thus, these two magnetically equivalent H_es were observed as a double doublet. By contrast, in **3a** (*cis*), the two H_as and two H_es adjacent to oxygens are magnetically equivalent and appeared as a singlet. This is most likely attributed to the rapid interconversion between the two unstable chair conformations, of which one of the two bulky groups must lie at the axial position. Product **3a** (*trans*), of which two bulky groups are equatorial, interacts more strongly with silica gel in TLC than **3a** (*cis*), which has a bulky axial group. The R_f values of **3a** (*trans*) and **3a** (*cis*) are 0.22 and 0.27, respectively. This, along with the higher melting point of **3a** (*trans*), is consistent with the stereochemical assignments based on ^1H NMR data (8).

TABLE 1
Yields and *trans/cis* Ratios of Bromides 3a–d

Compound	3a	3b	3c	3d
Yield (%)	74	61	68	67
trans/cis	—	2.6	1.2	4.3



a, R = $C_{12}H_{25}$; b, R = $C_{10}H_{21}$; c, R = C_8H_{17}; d, R = C_6H_{13}

SCHEME 1

TABLE 2
Yields, Melting Points, CMCs[a] and *trans/cis* Ratios of Surfactants 4a (*trans*), 4a (*cis*) and 4a–d

Compound	**4a** (*trans*)	**4a** (*cis*)	**4a**	**4b**	**4c**	**4d**
Yield (%)	63	60	81	62	75	86
Melting point (°C)	171–172	164–165	151–152	151–152	152–153	153–154
CMC (mM)	0.9	1.0	0.9	3.8	18	73
trans/cis	—	—	1.3	3.2	1.4	4.6

[a] CMC, Critical micelle concentration.

In refluxing triethylamine, **3a** (*trans*) and **3a** (*cis*) were converted to **4a** (*trans*) and **4a** (*cis*), respectively. The structural assignments for **4a** (*trans*) and **4a** (*cis*) were in accord with those for **3a** (*trans*) and **3a** (*cis*).

The *trans* and *cis* diastereomers and a mixture of the *trans* and *cis* diastereomers showed the same range of melting point (8). However, we observed that the melting point of **4a** (*trans*) was higher than that of **4a** (*cis*) (Table 2). This agrees with their conformation. Furthermore, the melting point of the recrystallized mixture of **4a** (*trans*) and **4a** (*cis*) was lower than that of either diastereomer. The lower melting point of the mixture of **4a** (*trans*) and **4a** (*cis*) originated from congruent melting of the recrystallized mixture.

The *trans*-to-*cis* ratios of bromides **3b-d** and surfactants **4a–d,** shown in Table 1 and Table 2, were determined by the integral intensity ratios of diagnostic hydrogens adjacent to oxygens and acetal hydrogen of each diastereomer.

Jaeger and co-workers (8) have also reported that each of two diastereomers and the mixture of them have the same CMC within experimental error. Additionally, their liability to acid showed no significant difference. At an early stage of this research, we found that **4a** (*trans*), **4a** (*cis*) and the mixture of them have the same CMC, too. Therefore, no attempt was made to further separate each of the two diastereomers of **3b–d** and **4b–d**. In the hydrolytic experiment, the mixture of **4a–d** was used.

TABLE 3
Analytical and Spectral Data of Bromides 3a (*trans*), 3a (*cis*) and 3b–d

	Elemental analysis found (calc'd)			
Compound	C (%)	H (%)	^{1}H NMR (400 MHz, $CDCl_3$-TMS, δ)	IR (cm^{-1})
3a (*trans*)	59.44 (59.50)	9.81 (9.71)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.03 (*m*, 2H, CH_2CH_3), 1.25 (*m*, 20H, $(CH_2)_{10}$), 1.96 (*m*, 1H, CH_2CH), 2.15 (*m*, 2H, CH_2CH_2Br) 3.32 (*t*, *J* = 11.5 Hz, 2H, H_a), 3.46 (*t*, *J* = 7 Hz, 2H, CH_2Br), 4.07 (*dd*, *J* = 11.5 Hz, 4.5 Hz, 2H, He), 4.62 (*t*, *J* = 5Hz, 1H, $OCHCH_2$)	2956, 2920, 2850, 1471, 1406, 1388, 1269, 1228, 1168, 1125, 1075, 1061, 1021, 991, 946, 884, 721, 643
3a (*cis*)	59.62 (59.50)	9.83 (9.71)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.26 (*M*, 21H, $(CH_2)_{10}$, CH_2CH), 1.68 (*m*, 2H, CH_2CH), 2.14 (*m*, 2H, CH_2CH_2Br), 3.46 (*t*, *J* = 7 Hz, 2H, CH_2Br), 3.91 (*s*, 4H, H_a, H_e), 4.71 (*t*, *J* = 5 Hz, 1H, $OCHCH_2$)	2957, 2922, 2853, 1464, 1405, 1378, 1264, 1240, 1152, 1133, 1022, 878, 721, 655
3b	57.32 (57.31)	9.43 (9.32)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.02, 1.25, 1.68, 1.96 (4*m*, 19H total, $(CH_2)_9$, CH_2CH), 2.15 (*m*, 2H, CH_2CH_2Br), 3.33 [*t*, *J* = 11.5 Hz, 1.44 H, H_a (*trans*)], 3.46 (*t*, *J* = 7 Hz, 2H, CH_2Br), 3.9 [(*s*, 1.12H, H_a, H_e (*cis*)], 4.07 [*dd*, *J* = 11.5 Hz, 4.5 Hz, 1.44H, H_e (*trans*)], 4.62 [*t*, *J* = 5 Hz, 0.72H, $OCHCH_2$(*trans*)], 4.71 [*t*, *J* = 5 Hz, 0.28H, $OCHCH_2$ (*cis*)]	2958, 2926, 2854, 1465, 1405, 1382, 1263, 1239, 1148, 1133, 1074, 1031, 991, 884, 723, 669, 650
3c	54.54 (54.72)	8.90 (8.86)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.02, 1.25, 1.68, 1.95 (4*m*, 15H total, $(CH_2)_7$, CH_2CH), 2.15 (*m*, 2H, CH_2CH_2Br), 3.32 [*t*, *J* = 11.5 Hz, 1.10 H, H_a (*trans*)], 3.46(*t*, *J* = 7 Hz, 2H, CH_2Br), 3.91 [*s*, 1.80H, H_a, H_e (*cis*)], 4.07 [*dd*, *J* = 11.5 Hz, 4.5 Hz, 1.10H, H_e (*trans*)], 4.62 [*t*, *J* = 5 Hz, 0.55H, OC HCH_2 (*trans*)], 4.71 [*t*, *J* = 5 Hz, 0.45H, $OCHCH_2$ (*cis*)]	2958, 2926, 2854, 1465, 1405, 1385, 1263, 1237, 1147, 1132, 1073, 1030, 991, 884, 723, 669, 647
3d	51.91 (51.62)	8.49 (8.30)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.03, 1.25, 1.69, 1.96 (4*m*, 11H total, $(CH_2)_5$, CH_2CH), 2.15 (*m*, 2H, CH_2CH_2Br), 3.33 [*t*, *J* = 11.5 Hz, 1.62 H, H_a (*trans*)], 3.46 (*t*, *J* = 7 Hz, 2H, CH_2Br), 3.91 [*s*, 0.76H, H_a, H_c (*cis*)], 4.07 [*dd*, *J* = 11.5 Hz, 4.5 Hz, 1.62H, H_e (*trans*)], 4.62 [*t*, *J* = 5 Hz, 0.81H, $OCHCH_2$(*trans*)], 4.71 [*t*, *J* = 5 Hz, 0.19H, $OCHCH_2$(*cis*)]	2958, 2926, 2854, 1465, 1405, 1382, 1264, 1240, 1149, 1134, 1072, 1031, 991, 884, 724, 669, 646

[a] NMR, nuclear magnetic resonance; TMS, trimethylsilyl; IR, infrared.

TABLE 4
Analytical and Spectral Data of Surfactants 4a (*trans*), 4a (*cis*) and 4a–d

Compound	Elemental analysis found (calc'd) C (%)	H (%)	N (%)	^{1}H NMR (400 MHz, $CDCl_3$-TMS, δ)	IR (cm^{-1})
4a (*trans*)	61.81 (62.05)	11.04 (10.85)	2.91 (3.01)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.02 (*m*, 2H, CH_2CH_3), 1.25 [*m*, 20H, $(CH_2)_{10}$], 1.39 (*t*, *J* = 7 Hz, 9H, CH_3CH_2N), 1.90 (*m*, 1H, CH_2CH), 2.07 (*m*, 2H, CH_2CH_2N), 3.32 (*t*, *J* = 11.5 Hz, 2H, H_a), 3.42 (*m*, 2H, CH_2CH_2N), 3.52 (*q*, *J* = 7 Hz, 6H, CH_3CH_2N), 4.02 (*dd*, *J* = 11.5 Hz, 4.5 Hz, 2H, H_e), 4.72 (*t*, *J* = 4 Hz, 1H, $OCHCH_2$)	2959, 2920, 2850, 1493, 1468, 1413, 1391, 1363, 1145, 1108, 1065, 1033, 1000, 942, 805, 721, 678
4a (*cis*)	61.82 (62.05)	11.01 (10.85)	3.11 (3.01)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.25 (*m*, 21H, $(CH_2)_{10}$, CH_2CH), 1.39 (*t*, *J* = 7 Hz, 9H, CH_3CH_2N), 1.61 (*m*, 2H, CH_2CH), 2.05 (*m*, 2H, CH_2 CH_2N), 3.41 (*m*, 2H, CH_2CH_2N), 3.53 (*q*, *J* = 7 Hz, 6H, CH_3CH_2N), 3.87 (*s*, 4H, H_a, H_e), 4.79 (*t*, *J* = 4 Hz, 1H, $OCHCH_2$)	2964, 2919, 2851, 1494, 1469, 1416, 1390, 1163, 1146, 1128, 1108, 1060, 1003, 920, 808, 719
4a	61.97 (62.05)	11.02 (10.85)	2.92 (3.01)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.02, 1.26, 1.61, 1.90 (4*m*, 23H total, $(CH_2)_{11}$, CH_2CH), 1.40 (*t*, *J* = 7 Hz, 9H, CH_3CH_2N), 2.05 (*m*, 2H, CH_2 CH_2N), 3.31 [*t*, *J* = 11.5 Hz, 1.14H, H_a (*trans*)], 3.41 (*m*, 2H, CH_2CH_2 N), 3.51, 3.52 (2*q*, *J* = 7 Hz, 7 Hz, 6H, CH_3CH_2N), 3.88 [*s*, 1.72H, H_a, H_e(*cis*)], 4.02 [*dd*, *J* = 11.5 Hz, 4.5 Hz, 1.14H, H_e (*trans*)], 4.71 [*t*, *J* = 4 Hz, 0.57H, $OCHCH_2$ (*trans*)], 4.79 [*t*, *J* = 4 Hz, 0.43H, $OCHCH_2$ (*cis*)]	2960, 2919, 2850, 1493, 1468, 1413, 1391, 1163, 1146, 1108, 1065, 1034, 1001, 942, 921, 806, 721, 677
4b	60.32 (60.53)	10.66 (10.62)	3.28 (3.21)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.01, 1.25, 1.60, 1.90 (4*m*, 19H total, $(CH_2)_9$, CH_2CH), 1.39 (*t*, *J* = 7 Hz, 9H, CH_3CH_2N), 2.04 (*m*, 2H, CH_2 CH_2N), 3.31 [*t*, *J* = 11.5 Hz, 1.52H, H_a (*trans*)], 3.42 (*m*, 2H, CH_2CH_2 N), 3.51, 3.52 (2*q*, *J* = 7 Hz, 7 Hz, 6H, CH_3CH_2N), 3.88 [*s*, 0.96H, H_a, H_e (*cis*)], 4.02 [*dd*, *J* = 11.5 Hz, 4.5 Hz, 1.52H, H_e (*trans*)], 4.71 [*t*, *J* = 4 Hz, 0.76H, $OCHCH_2$ (*trans*)], 4.79 [*t*, *J* = 4 Hz, 0.24H, $OCHCH_2$ (*cis*)]	2960, 2920, 2850, 1494, 1468, 1415, 1391, 1162, 1146, 1108, 1065, 1035, 1002, 938, 921, 806, 721, 676
4c	59.07 (58.81)	10.38 (10.36)	3.46 (3.43)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.02, 1.25, 1.61, 1.90 (4*m*, 15H total, $(CH_2)_7$, CH_2CH), 1.40 (*t*, *J* = 7 Hz, 9H, CH_3CH_2N), 2.05 (*m*, 2H, CH_2 CH_2N), 3.31 [*t*, *J* = 11.5 Hz, 1.18H, H_a (*trans*)], 3.42 (*m*, 2H, CH_2CH_2 N), 3.51, 3.52 (2*q*, *J* = 7 Hz, 7 Hz, 6H, CH_3CH_2N), 3.88 [*s*, 1.64H, H_a, H_e (*cis*)], 4.02 [*dd*, *J* = 11.5 Hz, 4.5 Hz, 1.18H, H_e (*trans*)], 4.71 [*t*, *J* = 4 Hz, 0.59H, $OCHCH_2$ (*trans*)], 4.79 [*t*, *J* = 4 Hz, 0.41H, $OCHCH_2$ (*cis*)]	2961, 2919, 2851, 1494, 1468, 1417, 1391, 1163, 1146, 1107, 1065, 1031, 1002, 942, 921, 806, 720, 676
4d	57.09 (56.83)	10.23 (10.07)	3.74 (3.68)	0.88 (*t*, *J* = 7 Hz, 3H, CH_3), 1.02, 1.25, 1.61, 1.90 (4*m*, 11H total, $(CH_2)_5$, CH_2CH), 1.39 (*t*, *J* = 7 Hz, 9H, CH_3CH_2N), 2.04 (*m*, 2H, CH_2 CH_2N), 3.31 (*t*, *J* = 11.5 Hz, 1.64H, H_a (*trans*)], 3.42 (*m*, 2H, CH_2CH_2 N), 3.51, 3.52 (2*q*, *J* = 7 Hz, 7 Hz, 6H, CH_3CH_2N), 3.88 [*s*, 0.72H, H_a, H_e (*cis*)], 4.02 [*dd*, *J* = 11.5 Hz, 4.5 Hz, 1.64H, H_e (*trans*)], 4.71 [*t*, *J* = 4 Hz, 0.82H, $OCHCH_2$ (*trans*)], 4.79 [*t*, *J* = 4Hz, 0.18H, $OCHCH_2$ (*cis*)]	2959, 2923, 2853, 1493, 1468, 1416, 1390, 1163, 1147, 1107, 1065, 1029, 1001, 940, 921, 805, 723, 677

[a] See Table 3 for abbreviations.

Surfactants **4a** (*trans*), **4a** (*cis*) and **4a–d** have good water solubility at room temperature. The Krafft points of **4a** (*trans*) and **4a** (*cis*) were 6 and 7°C, respectively, and those of **4a–d** were lower than 0°C. The increase in hydrophilicity of surfactants **4a–d**, compared to conventional surfactants with comparable length of alkyl chain, is attributed to the introduction of two acetal oxygens of 1,3-dioxane ring (24). The good water solubility and low Krafft points make them applicable in a wide range of micelle study.

Surfactant **4a–d** can be decomposed under acidic conditions to give corresponding 2-alkyl-1,3-propanediol and another water-soluble nonsurfactant **5** (Scheme 2).

The destructibility of surfactants **4a–d** in 2 N HCl at 50°C is shown in Table 5. As observed for surfactants with a 1,3-dioxolane ring (11–13), under the same conditions, the hydrolytic reactivity of surfactants **4a–d** decreased with increasing alkyl chainlength, and the time needed to completely decompose surfactants **4a–d** was longer than that of corre-

$$4 \xrightarrow[H_2O]{H^+} \underset{\mathbf{1}}{RCH(CH_2OH)_2} + \underset{\mathbf{5}}{CHOCH_2CH_2N^+(CH_2CH_3)_3Br^-}$$

SCHEME 2

TABLE 5
Destructibility of Surfactants 4a–d in 2N HCl at 50°C

Compound	4a		4b		4c		4d	
Surfactant concentration (mM)	10	20	10	20	10	20	10	20
Time (h)	70	90	60	70	50	60	35	45
% Decomposition	87	93	100	90	100	100	100	100

sponding anionic surfactants with the same length of alkyl chain (19).

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