Synthesis and Surface Properties of Chemodegradable Anionic Surfactants: Sodium (2-*n*-Alkyl-1,3-Dioxan-5-yl)Sulfates[1]

Andrzej Piasecki*, Adam Sokołowski, Bogdan Burczyk, and Urszula Kotlewska
Institute of Organic and Polymer Technology, Technical University of Wrocław, 50-370 Wrocław, Poland

ABSTRACT: In the reaction of *cis*- and *trans*-2-*n*-alkyl-5-hydroxy-1,3-dioxane mixtures with $SO_3 \cdot$ pyridine complex, followed by neutralization with sodium hydroxide or sodium carbonate, a new group of anionic surfactants, i.e., sodium *cis*- and *trans*-(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates were obtained. The hydrophobic intermediates used in the sulfation reaction were obtained in high yields from four-component glycerol acetals by the process of transacetalization and selective crystallization of 1,3-dioxane derivatives. The physical data of the new compounds and some of their surface properties, such as critical micelle concentration (CMC), effectiveness of water surface tension reduction Π_{CMC}, standard free energies of adsorption and micellization, ΔG^o_{ads} and ΔG^o_{CMC}, surface excess concentration, Γ_{CMC}, and the surface area demand per molecule, A_{CMC}, were determined. It was shown that the surface activity of the standard anionic surfactant sodium dodecyl sulfate should be similar to the surface activity of sodium (2-*n*-decyl-1,3-dioxan-5-yl)sulfate.

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KEY WORDS: Adsorption, cyclic acetal-type anionic surfactants, micellization, sodium (2-*n*-alkyl-1,3-dioxan-5-yl)sulfate.

Glycerol acetals, i.e., mixtures of *cis*- and *trans*-2-alkyl-4-hydroxymethyl-1,3-dioxolanes and *cis*- and *trans*-2-alkyl-5-hydroxy-1,3-dioxanes (1), were first used as hydrophobic intermediates in nonionic surfactant synthesis by Rutzen and Goette (2). However, in this mixture of cyclic acetals, 1,3-dioxolane derivatives bear a primary hydroxyl group, whereas the 1,3-dioxane derivatives bear a secondary one. This is inconvenient for further reactions because of the different reactivities of the hydroxyl groups in question. We were the first to synthesize oxyethylenated 2-alkyl-4-hydroxymethyl-1,3-dioxolanes, the starting materials being obtained in a reaction of glycerol α-monoacetate with aliphatic aldehydes (3,4). Later, Jaeger *et al.* (5) and Ono *et al.* (6) synthesized several glycerol-based surfactants with a 1,3-dioxolane ring in the surfactant's molecules. To obtain surface-active 1,3-dioxane derivatives, there were two possible routes: either a rather difficult isolation of 2-alkyl-5-hydroxy-1,3-dioxanes from a four-component glycerol acetal mixture, or an acetalization procedure with aliphatic aldehydes and other polyols that contain at least three hydroxyl groups in their molecules. Thus, starting with 2,2-*bis*(hydroxymethyl)-1,3-propanediol, 2-alkyl-5,5-*bis*(hydroxymethyl)-1,3-dioxanes were obtained and further reacted with oxirane or 1,3-propane sultone to yield nonionic (7) or sulfonate salt-type anionic (8) surfactants, respectively. In a reaction of 1,1,1-*tris*(hydroxymethyl)propane and 1,1,1-*tris*(hydroxymethyl)ethane with aliphatic aldehydes, 2-*n*-alkyl-5-hydroxymethyl-5-ethyl-1,3-dioxanes and 2-*n*-alkyl-5-hydroxymethyl-5-methyl-1,3-dioxanes were synthesized as hydrophobic intermediates for the preparation of oxyethylenated nonionic (9) and sulfate salt-type anionic (10) surfactants, respectively. The reaction of aldehydes with diethyl 2,2-*bis*(hydroxymethyl)malonate yielded dianionic surfactants with two carboxy groups at the C-5 atom of the 1,3-dioxane ring (11), whereas in a condensation reaction of 2-alkyl-1,3-propanediols with 3-bromopropanal, followed by a reaction with triethylamine, cationic surfactants with a 1,3-dioxane ring were prepared (12).

Recently, we devised a convenient and simple method of isolation of long-chain *cis*- and *trans*-2-alkyl-5-hydroxy-1,3-dioxanes from a four-component mixture of glycerol acetals (13,14). This procedure gives easy access to hydrophobic 1,3-dioxane derivatives that contain a secondary hydroxyl group. In this paper, we report the synthesis and basic surface properties of new, chemodegradable anionic surfactants of the sulfate salt-type, i.e., sodium (2-*n*-alkyl-1,3-dioxan-5-yl)sulfates. Using straight-chain aliphatic aldehydes **1a–c** (*n*-octanal, *n*-decanal, and *n*-dodecanal) and glycerol **2**, we obtained four-component mixtures of **3a–c** and **4a–c**, which were transformed into two-component mixtures of *cis*- and *trans*-2-*n*-alkyl-5-hydroxy-1,3-dioxanes **4a–c** with high yields (Scheme 1). These intermediates were subjected to sulfation reactions with $SO_3 \cdot$ pyridine complex, followed by neutralization, to obtain mixtures of sodium *cis*- and *trans*-(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates, **5a–c**. We determined the physicochemical and surface properties of the surfactants under study.

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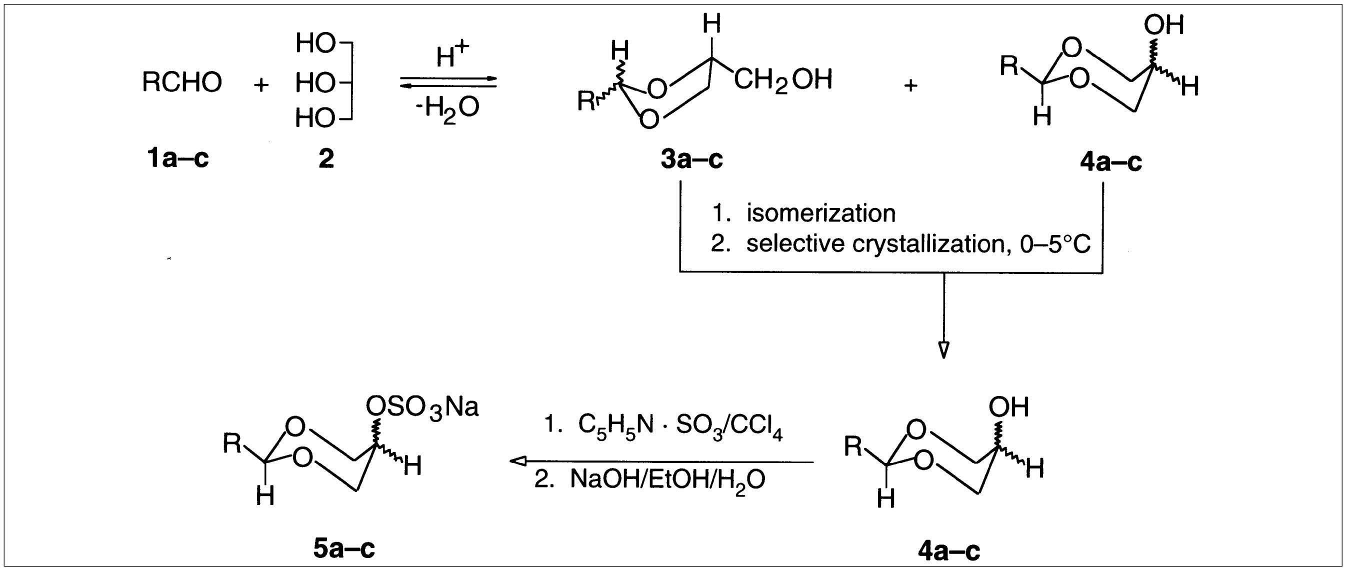
*To whom correspondence should be addressed at Institute of Organic and Polymer Technology, Technical University of Wrocław, Wybrzeże Stanisława Wyspiańskiego 27, 50-370 Wrocław, Poland.



SCHEME 1

MATERIALS AND METHODS

Materials. n-Octanal, *n*-decanal, and *n*-dodecanal (Merck, Germany) were used after purification by distillation and stabilization with 0.1% by weight hydroquinone. Glycerol, pyridine (POCh, Poland), and chlorosulfonic acid (Merck) were used as received. All reagents were of analytical grade.

Synthesis of cis- *and* trans-*2-*n*-alkyl-5-hydroxy-1,3-dioxanes* **4a–c**. 0.1 Mole of the four-component mixture of glycerol acetals, obtained from aliphatic aldehydes **1a–c** and glycerol **2** by a known procedure (1,15), i.e., a mixture of *cis*- and *trans*-2-*n*-alkyl-4-hydroxymethyl-1,3-dioxolanes **3a–c** and *cis*- and *trans*-2-*n*-alkyl-5-hydroxy-1,3-dioxanes **4a–c**, in 50 mL hexane was maintained for 2 d at room temperature and then for 5 d at 0–5°C in the presence of 0.1 g of *p*-toluenesulfonic acid monohydrate. The precipitated mixture of *cis*- and *trans*-2-*n*-alkyl-5-hydroxy-1,3-dioxanes **4a–c** was then filtered off and recrystallized twice from hexane (70–90% mol yield) (13,14).

Synthesis of sodium cis- *and* trans-*(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates* **5a–c**. Freshly prepared sulfur trioxide · pyridine complex (8 g-0.06 mole) (10,16) was introduced gradually at room temperature to a well-stirred solution of 0.05 mole of **4a–c** in 100 mL of dry carbon tetrachloride and 3–5 mL of dry pyridine. The reaction was continued for 1 h at room temperature and then for 6–8 h at 45°C. After concentration under reduced pressure to half of the original volume, the reaction mixture was added drop by drop to a cooled solution of 0.065 mole NaOH in 150 mL ethanol and 50 mL water. The solvents were evaporated under reduced pressure, and the solid residue was washed and filtered several times with 50 mL of boiling ethanol. Combined ethanol filtrates were concentrated, and after cooling, a mixture of sodium *cis*- and *trans*-(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates **5a–c** was crystallized as white needles. The mother liquor was then concentrated, and an additional portion of **5a–c** was obtained. After recrystallization from dry ethanol or methanol, a mixture of sodium *cis*- and *trans*-(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates was obtained with 91–93 mol% yield (17).

Analytical methods. ^{1}H nuclear magnetic resonance (NMR) spectra were measured for solutions of **4a–c** in $CDCl_3$ with tetramethylsilane as a standard and for solutions of **5a–c** in D_2O on a Bruker Avance DRX300 spectrometer. The gas–liquid chromatography (GLC) was performed with a Giede GCHF-18.3 apparatus (Giede, Germany), equipped with a 5% Silicon XE60 on GasChrom Q, 80/100 mesh, packed column (1 m length). Nitrogen was used as carrier gas. Surface tension measurements were performed with a K12e (Krüss, Germany) processor tensiometer on surfactant solutions in 10^{-4} M $NaHCO_3$ in triple-distilled water at 31°C. Surface tension measurements and elemental analyses data were reproducible within ±0.02 mN/m and ±0.05 wt%, respectively.

RESULTS AND DISCUSSION

Preparation, physical properties, and stereoisomer composition of hydrophobic intermediates. Hydrophobic intermediates constituted mixtures of geometric isomers of *cis*- and *trans*-2-*n*-alkyl-5-hydroxy-1,3-dioxanes **4a–c** and were obtained in high yields from four-component mixtures of glycerol acetals, **3a–c** and **4a–c**, produced from the reaction between aliphatic aldehydes **1a–c** and glycerol **2** (Scheme 1). This procedure was comprised of a transacetalization reaction of glycerol acetals and selective crystallization of 1,3-dioxane derivatives **4a–c** from hexane solution. Crude precipitates contained at least 96 mol% of 1,3-dioxane derivatives. Recrystallization from hexane leads to pure mixtures of *cis*- and *trans*-2-*n*-alkyl-5-hydroxy-1,3-dioxanes **4a–c** as low-melting solids with physicochemical properties and composition as presented in Table 1. GLC of **4a–c** mixtures showed the presence of two components with considerably different

TABLE 1
Physiochemical Properties of Mixtures of *cis*- and *trans*-2-*n*-Alkyl-5-Hydroxy-1,3-Dioxanes 4a–c

	Yield (mol%)			
Compound	Crude product[a,b]	Recryst. product[a]	Melting point[c] (°C)	Diastereomer molar ratio[d] *cis*:*trans*
4a	57.8	50.0	28.5–47	59:41
4b	62.3	55.0	42.5–58	57:43
4c	88.0	83.0	50.5–65	63:37

[a] In relation to four-component mixture of glycerol acetals, **3a–c** and **4a–c**, used in the transacetalization/crystallization process.
[b] Precipitates of crude products contained 96.0, 98.6, and 99.0 mol% of 1,3-dioxane derivatives for **4a**, **4b**, and **4c**, respectively.
[c] Limited solubility of higher-melting *trans* isomer in a molten *cis* isomer is the reason for wide range of isomer mixture melting temperatures.
[d] Determined by gas–liquid chromatography; Recryst., recrystallized.

retention times. The determined relative retention times of *cis* and *trans* isomers were equal to *cis*:*trans* ≅ 0.38:1 for each pair of isomers. ^{1}H NMR analysis of mixtures of *cis*- and *trans*-2-*n*-alkyl-5-hydroxy-1,3-dioxanes showed the presence of two 2,5 disubstituted-1,3-dioxane derivatives with equatorial alkyl radicals located at C-2 and an axial hydroxyl group –OH in *cis* isomers and an equatorial hydroxyl group in *trans* isomers located at the C-5 carbon atom of the 1,3-dioxane ring. The axial protons $H_{(a)}$-2 at C-2 absorb as triplets ($J = 5.2$ Hz) at $\delta = 4.55$ ppm in *cis* isomers and at $\delta = 4.40$ ppm in *trans* isomers. The equatorial proton $H_{(e)}$-5 at the C-5 carbon atom in *cis* isomers absorbs as a multiplet ($J = 1.5$ Hz) at $\delta = 3.51$ ppm, while the axial $H_{(a)}$-5 proton in *trans* isomers absorbs as a heptet ($J = 5.2$ and 10.4Hz) at $\delta = 3.87$ ppm.

Physical and surface properties of mixtures of sodium cis- *and* trans-*(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates* **5a–c**. The method of sulfation of acetal-type compounds with $SO_3 \cdot$ pyridine complex, which was successfully used by us for the preparation of sodium [(2-*n*-alkyl-5-methyl-1,3-dioxan-5-yl)methyl]sulfates from 2-*n*-alkyl-5-hydroxymethyl-5-methyl-1,3-dioxanes with a primary hydroxyl group (10,16), was also efficient for the sulfation of compounds **4a–c** with a secondary hydroxyl group (17) after a slight modification of the period and temperature of the reaction. The physical properties of mixtures of sodium *cis*- and *trans*-(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates **5a–c**, which were prepared with yields that exceeded 90 mol% for the recrystallized products, are presented in Table 2. Because of the high yield of sulfation reactions and crystallization procedures, the compositions of mixtures of surface-active sodium *cis*- and *trans*-(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates **5a–c** were almost equal to the compositions of initial **4a–c**. Elemental analysis of prepared compounds **5a–c** revealed their molecular formulae to be as presented in Table 2. ^{1}H NMR analyses of **5a–c** have shown that the sulfation reaction proceeds with retention configuration, i.e., from *cis*-**4a–c,** *cis*-**5a–c** were obtained, and analogously, *trans*-**4a–c** produced *trans*-**5a–c**, respectively. ^{1}H NMR spectra recorded for solutions of sodium sulfates **5a–c** in D_2O (the chemical shift of protons of H_2O, present as impurity in D_2O, was stabilized at $\delta = 4.700$ ppm) are similar to ^{1}H NMR spectra of their hydrophobic precursors. The axial protons $H_{(a)}$-2 at C-2 absorb as triplets ($J = 5$ Hz) at $\delta = 4.62$ ppm in *cis* isomers and at $\delta = 4.43$ ppm in *trans* isomers. The equatorial protons $H_{(e)}$-5 in *cis* isomers absorb as multiplets at $\delta = 4.28$ ppm ($J = 1.5$ Hz), while $H_{(a)}$-5 protons in *trans* isomers absorb as heptets at $\delta = 4.34$ ppm ($J = 5.2$ and 10.4 Hz). The equatorial protons $H_{(e)}$-4,6, located at C-4 and C-6 carbon atoms of the 1,3-dioxane ring in both **4a–c** and **5a–c** molecules, absorb as double doublets at $\delta \cong 4.0 \div 4.2$ ppm ($J = 1.5$ and 12.6 Hz) in *cis* isomers and at $\delta \cong 4.2$ ppm ($J = 5.2$ and 10.4 Hz) in *trans* isomers. The axial protons $H_{(a)}$-4,6 absorb as double doublets at $\delta \cong 3.8 \div 4.0$ ppm ($J = 1.5$ and 12.5 Hz) in *cis* isomers of **4a–c** and **5a–c**, while in *trans* isomers of **4a–c** and **5a–c** they absorb as triplets at $\delta \cong 3.4 \div 3.5$ ppm ($J = 10.4$ Hz).

The Krafft point values [determined by observation of the temperatures at which crystallization of **5a–c** from their aqueous solutions took place (Table 3)] are higher than those determined for mixtures of sodium *cis*- and *trans*-[(2-*n*-alkyl-5-methyl-1,3-dioxan-5-yl)methyl]sulfates (10).

The surface tensions γ of aqueous solutions of the surfactants under study at 31°C are plotted against the logarithms of the concentrations in Figure 1, which also includes the sodium dodecyl sulfate isotherm (18). As we expected, the surface activity of anionic surfactants **5a–c** rapidly increases as the length of the alkyl chain R increases. From the location of the isotherms, it can be expected that the surface activity of sodium dodecyl sulfate is similar to the surface activity of the 1,3-dioxane derivative that contains an *n*-decyl radical $C_{10}H_{21}$ at the C-2 carbon atom of the 1,3-dioxacyclane ring. The surface properties of sodium sulfates **5a–c** are presented in Table 3. Based on the Gibbs adsorption equation for a 1:1 electrolyte (19), the surface excess Γ_{CMC} and the surface area demand per molecule at the adsorption layer A_{CMC} were determined from

TABLE 2
Physicochemical Properties of Mixtures of Sodium *cis*- and *trans*-(2-*n*-Alkyl-1,3-Dioxan-5-yl)Sulfates 5a–c

					Elemental analysis found (calcd.) (%)		
Compound	R	Molecular formula	Yield[a] (mol%)	Melting point[b] (°C)	C	H	S
5a	n-C_7H_{15}	$C_{11}H_{21}SO_6Na$	92.0	200–202	43.4 (43.4)	7.0 (7.0)	10.5 (10.5)
5b	n-C_9H_{19}	$C_{13}H_{25}SO_6Na$	93.0	196–200	47.0 (47.0)	7.5 (7.6)	9.7 (9.6)
5c	n-$C_{11}H_{23}$	$C_{15}H_{29}SO_6Na$	90.5	198–200	50.0 (50.0)	8.0 (8.1)	9.0 (8.9)

[a] Recrystallized product. [b] With decomposition.

TABLE 3
Surface Properties of Mixtures of Sodium *cis*- and *trans*-(2-*n*-Alkyl-1,3-Dioxan-5-yl)Sulfates 5a–c

Compound	Krafft point (°C)	CMC · 10^3 (mol/dm^3)	$\Gamma_{CMC} \cdot 10^6$ (mol/m^2)	$A_{CMC} \cdot 10^{20}$ (m^2)	Π_{CMC} (mN/m)	ΔG^o_{CMC} (kJ/mol)	ΔG^o_{ads} (kJ/mol)
5a	—	64.7	3.1_3	$53._0$	29.7	-32.3_8	-55.1_4
5b	18	13.3	3.3_5	$49._6$	30.1	-38.9_7	-63.7_2
5c	30	3.62	3.2_4	$51._3$	32.0	-44.6_7	-72.0_6

the $\gamma = f(\log c)$ plot for points near the CMC. The Γ_{CMC} values for **5a–c** oscilate slightly near $3.2 \cdot 10^{-6}$ mol/m^2 ($A_{CMC} \approx 51 \cdot 10^{-20}$ m^2). It suggests that the orientation of **5a–c** molecules at the adsorption layer is fixed, independent of the length of the R radical. The surface areas A_{CMC} of **5a–c** sulfates are considerably higher than that of sodium dodecyl sulfate, probably because of the presence of the bulky 1,3-dioxane ring in their molecules. However, those areas are much lower than the surface areas determined for sodium [(2-*n*-alkyl-5-methyl-1,3-dioxan-5-yl)methyl]sulfates (10), which contain two geminally located substituents at the C-5 carbon atom of the 1,3-dioxane ring. The effectiveness of water surface tension reduction by **5a–c** increases as the alkyl chain R increases and is higher than the effectiveness of 2-*n*-alkyl-5-hydroxymethyl-5-methyl-1,3-dioxane derivatives. The logarithm of the CMC (log CMC) of compounds **5a–c** is a linear function of the R-radical length with a slope equal to –0.3 (correlation coefficient 0.998). This slope is characteristic for common anionic surfactants without added electrolyte, e.g., sodium alkyl carboxylates or sodium alkyl sulfates, –0.30 (20). Likewise, the standard free energies of micellization, ΔG^o_{CMC}, calculated according to Equation 1 (21,22):

$$\Delta G^o_{CMC} = (2 - \alpha)RT\ln x_{\pm CMC} \qquad [1]$$

where $x_{\pm CMC} = x_{CMC} \cdot y_\pm$ is the standard mean activity in mole fraction units at the CMC ($y_\pm$ is the mean activity coefficient, $\log y_\pm = -0.509J^{0.5}$; J is the ionic strength), and α is the micellar degree of dissociation [we assume $\alpha = 0.18$, as it was determined for sodium alkyl sulfates (23)], and the standard free energy of adsorption according to Equation 2:

$$\Delta G^o_{ads} = -2\ RT\ln B \qquad [2]$$

where B is an equilibrium constant of the Temkin isotherm adsorption (24,25). Both are linear functions of the length of alkyl radical R.

Values of the increments of the standard free energy of micellization and the standard free energy of adsorption, calculated for one methylene group $-CH_2-$ of R radical, $\Delta G^o_{CMC}/CH_2$ and $\Delta G^o_{ads}/CH_2$, are equal to -3.3_6 and -4.2_4 (kJ/mol), respectively, with correlation coefficients higher than 0.999.

Aqueous solutions of sulfates **5a–c** are stable, and changes of their surface tensions were not observed over a period of more than one week.

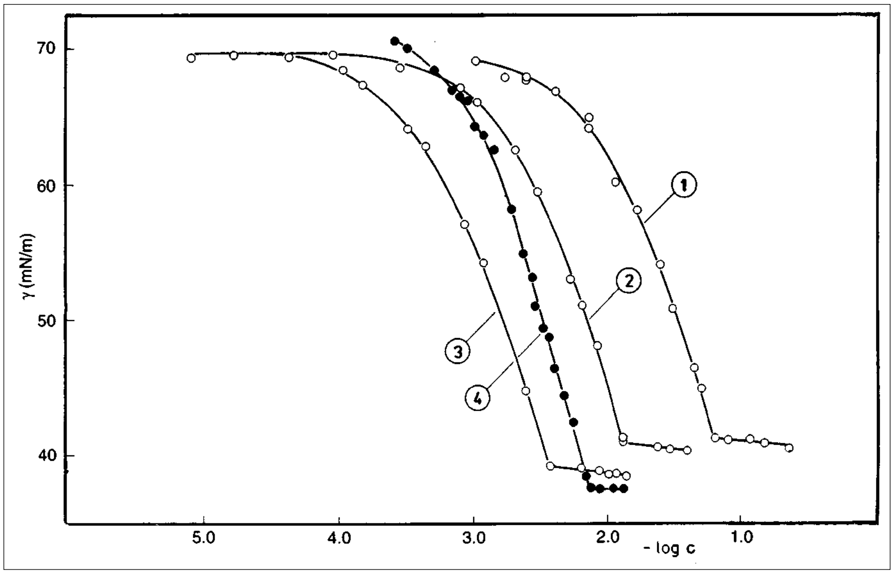


FIG. 1. The surface tension (γ) vs. logarithm of molar concentration (log c) of sodium *cis*- and *trans*-(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates at 31°C: 1, **5a**; 2, **5b**; 3, **5c**; 4, sodium dodecyl sulfate at 30°C [data from (Ref. 18)].

Because of (i) the simple method of hydrophobic intermediate preparation, (ii) the high yield of the sulfation reaction, and (iii) the high surface activity and stability in aqueous solutions, mixtures of sodium *cis*- and *trans*-(2-*n*-alkyl-1,3-dioxan-5-yl)sulfates seem to be an interesting new group of anionic surfactants that make efficient glycerol utilization possible.

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