

Scientific paper

Scalable Synthesis of Salt-free Quaternary Ammonium Carboxylate Catanionic Surfactants

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Abstract

Surfactants in commercial products commonly contain catanionic mixtures thus many studies of aqueous surfactant mixtures have been carried out. However, hardly any studies have been dedicated to pure catanionic surfactants often termed salt-free catanionic surfactants. One of the difficulties is in acquirement of samples with required purity due to difficult separation of these compounds from inorganic salts. In this work we present an alternative method of synthesis using dimethyl carbonate as the alkylating agent in order to obtain alkyl trimethylammonium alkanecarboxylates with medium alkyl chain lengths (6–10).

Keywords: Synthesis; surfactants; salt-free; catanionics; quaternization

1. Introduction

Commercial surfactants are commonly a mixture of cationic, anionic and non-ionic surfactants due to their enhanced performance as mixtures.^{1,2} Therefore, along the studies of non-ionic and ionic surfactants in their pure form as well as in binary aqueous solutions, significant focus has been dedicated to aqueous mixtures of cationic and anionic surfactants – catanionic mixtures. An interesting group of catanionic surfactants are salt-free catanionics, where often the challenge of preparing these surfactants in their pure form is the removal of all inorganic salts upon mixing the parent cation and anion salts. There are three main methods to remove inorganic salts from equimolar catanionic mixtures: (1) ion-exchange columns are used to prepare acids and hydroxides and subsequently mixed; (2) liquid-liquid extraction in organic phase and (3) precipitation method.³ When surfactants are poorly soluble in organic solvents precipitation is generally easily achieved. On the contrary, if solubility is limited in polar solvents liquid-liquid extraction is possible. However, many surfactants, especially those interesting for application, are soluble in both, organic and aqueous media. This limits the separa-

tion techniques to ion-exchange columns or the precipitation of silver halides from water solutions. Unfortunately, these two approaches are not economically viable and some contamination with inorganic salt can still occur. This can have significant impact on some of the surfactant's properties and can crucially affect studied physical and aggregation properties of aqueous solutions.^{4–6} Thus, the development of novel and more effective methods of synthesis is required.

In this work we present two synthetic procedures to prepare alkyltrimethylammonium alkanecarboxylate catanionic surfactants (Figure 1). Generally, the synthesis of quaternary ammonium surfactants starts with ammonia or substituted amine precursor. It is alkylated in two or more steps with the last step being quaternization. Most studied and produced quaternary ammonium surfactants are alkyltrimethylammonium halides followed by dialkyldimethylammonium halides (gemini surfactants).⁷ Their aqueous solutions are generally more stable than quaternary ammonium surfactants with remaining hydrogen(s) on quaternary ammonium. Quaternization can be achieved by the Menshutkin reaction where tertiary amine is reacted with haloalkane. However, halide free methods are preferred for industrial application.

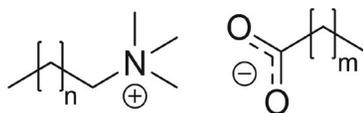


Figure 1. N-alkyl-N,N,N-trimethylammonium alkanecarboxylates.

Recently, Jiang *et al.* presented a synthetic procedure for the preparation of salt-free quaternary ammonium carboxylates using dimethyl carbonate (DMC) as alkylating agent of appropriate tertiary amines, with one or two alkyl chains exceeding 12 carbon atoms and a short-chain carboxylate anion (acetate, propionate, lactate).⁸ They expanded the scope to long-chain cationic surfactants in the follow-up papers, namely tetradecyltrimethylammonium alkanecarboxylates (hexanoate, octanoate, decanoate, dodecanoate, tetradecanoate)⁹ and alkyl (decyl, dodecyl, tetradecyl, hexadecyl, octadecyl) trimethylammonium decanoates.¹⁰ DMC represents an attractive eco-friendly alternative to methyl halides.¹¹

However, additional challenges emerge when both alkyl chains are shorter than 10 carbon atoms. Mainly, due to increased solubility in majority of polar and non-polar solvents the usual purification methods of precipitation, recrystallization and liquid-liquid extraction become less efficient or impossible. Additionally, the commercial price of tertiary amines, required in the most common synthesis approach, increases with shorter chains. Therefore, in this work we present low cost synthetic alternative starting with a secondary amine to produce quaternary ammonium carboxylate surfactants with alkyl chains between 6 and 10 carbon atoms long.

Synthesis of decyltrimethylammonium alkanecarboxylates (acetate, butanoate, hexanoate, octanoate, nonanoate, decanoate, undecanoate) has also been reported by reacting quaternary ammonium halides with silver hydroxide. After precipitation of the silver halide salts, the resulting hydroxide solutions were reacted with carboxylic acid.¹² For large scale application silver salts are not economically viable, therefore we present a competitive approach through precipitation of KCl from ethanol. We compare the thermal properties of products obtained by both procedures.

2. Experimental

2.1. General Information

The reagents and solvents in general procedures were used as obtained from the commercial sources (Merck), unless noted otherwise. Octanoic acid (**4b**) and decanoic acid (**4c**) were recrystallized from ethanol by addition of acetone. IR spectra were obtained with a Perkin-Elmer Spectrum 100, equipped with a Specac Golden Gate Diamond ATR as a solid sample support. High resolution mass spectra (HRMS) were recorded on Agilent 6224 time-of-flight (TOF) mass spectrometer equipped with a double orthogo-

nal electrospray source at atmospheric pressure ionization (ESI) coupled to an HPLC instrument. ¹H, ¹³C and ¹⁵N NMR spectra were recorded with a Bruker Avance III 500 MHz NMR (at 500 MHz, 126 MHz and 51 MHz, respectively) instrument at 296 K in DMSO-*d*₆ using TMS as an internal standard. Proton and carbon spectra were referenced to the residual chloroform shifts of 7.26 ppm and 77.16 ppm, respectively.¹³ Assignments of proton, carbon and nitrogen resonances were performed by 2D NMR techniques (¹H-¹H *gs*-COSY, ¹H-¹³C *gs*-HSQC, ¹H-¹³C *gs*-HMBC and ¹H-¹⁵N *gs*-HMBC). Carbon resonances without labels belong to the chain carbons and could not have been differentiated. Thermogravimetric (TG) measurements were performed on a Mettler Toledo TGA/DSC1 Instrument in the temperature range from 25 to 400 °C under dynamic air flow (100 cm³ min⁻¹) with a heating rate of 5 K min⁻¹. Approximately 3–5 mg of sample was weighed into a 150 μL platinum crucible and the baseline was subtracted. Differential scanning calorimetry (DSC) measurements were performed separately on a Mettler Toledo DSC 1 Instrument in 40 μL aluminium crucibles under the same conditions.

2.2. Synthesis

Alkyltrimethylammonium Chlorides 3a–c

In a 200 mL autoclave chloroalkanes **2a–c** (0.21 mol, 1 eq) were mixed with 33% trimethylamine solution in ethanol (**1**, 100 mL, 2 eq). The mixture was stirred in an oil bath at 80 °C for 3 days. Excess **1** and solvents were removed under reduced pressure. Products were dissolved in minimal amount of ethanol required (approx. 5 mL) and recrystallized by addition of ethyl acetate (100 mL). The precipitates were filtered and washed with diethyl ether (50 mL) and subsequently dried under reduced pressure to yield products in 50% average yield. Products **3** are very hygroscopic which reduces the yield of recrystallization in open air (**3a** 26%, **3b** 53%, **3c** 55%).

Alkyltrimethylammonium Carboxylates 5a–e According to Procedure A

In the next step, alkyltrimethylammonium chlorides **3a–c** as prepared above (0.07 mol) were dissolved in ethanol (20 mL) and weighed precisely. The exact amount of chloride ions was determined by AgNO₃ titration of small samples and the required equimolar amounts of carboxylic acids **4a–c** were weighed and added. 95% of KOH, required for neutralization, was weighed as solid pellets. After all of the added solid KOH dissolved and KCl precipitated (1 hour), the remaining KOH was titrated as an approximate 0.25 M solution of KOH in ethanol until potential of glass electrode dropped below –250 mV which was previously determined as the potential of the inflection point. Solutions were filtered to separate the filtrate containing the products from the precipitated KCl. Filtrates were concentrated under reduced pressure subsequently precipitating more KCl which was filtered to obtain filtrate

containing desired alkyltrimethylammonium carboxylates **5a–e** in quantitative yields. Solutions were dried first under reduced pressure and followed by high vacuum. However, due to increased solubility of KCl in the presence of the products up to 3% of KCl remains in the final product as determined by AgNO₃ titration and TG analysis.

Alkyldimethylamines **8a–c**

Bromoalkanes **7** (bromohexane (**7a**) 2.3 mol; bromooctane (**7b**) 1.9 mol; bromodecane (**7c**) 1.6 mol; 1 eq) were mixed with toluene (300 mL) in a 3 L autoclave. Then 40% *N,N*-dimethylamine (**6**) solution in water (500 mL, approx. 2 eq) was added. An excess amount of NaOH (200 mL) was added as a 50% aqueous solution. The mixtures were stirred and heated to 60 °C for 3 days. After the reaction was completed the reaction mixture separated in two layers. The organic phase containing products was separated from water phase and concentrated under reduced pressure. The products were washed several times with water (5 × 20 mL) and distilled under reduced pressure to ensure separation from inorganic byproducts and to remove toluene (**8a** 70%, **8b** 90%, **8c** 95%). While amine **8c** can be dried on a vacuum line with minimal loss due to evaporation, the distillation results in better overall yield. The presence of toluene in product is not problematic, as it is removed in the following steps.

Alkyltrimethylammonium Methylcarbonates **9a–c**

In a 50 mL autoclave alkyldimethylamines **8a–c** as prepared above (0.2 mol, 1 eq) were mixed with dimethyl carbonate (Me₂CO₃, 0.3 mol, 1.5 eq) and methanol (20 mL) as solvent. Mixtures were stirred at 120 °C for 2 days or 110 °C for 3 days. Excess Me₂CO₃ and solvent were removed under reduced pressure. Alkyltrimethylammonium methyl carbonates with traces of hydrogencarbonate analogues were recrystallized from ethyl acetate. The pure products **9** were obtained after recrystallization (**9a** 50%, **9b** 70%, **9c** 90%). Yields are primarily lowered by loss during purification however it should be noted that recrystallization in this step is not strictly necessary as the organic impurities are also successfully removed by recrystallization in the final step of the procedure B thus potentially increasing yield.

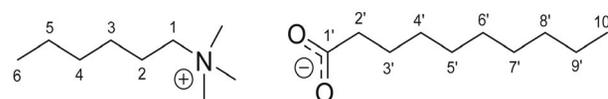
Alkyltrimethylammonium Carboxylates **5a–f** According to Procedure B

In a round-bottomed flask alkyltrimethylammonium methylcarbonates **9a–c** (0.03 mol) and carboxylic acids **4** (0.03 mol) were weighed. Small amount of methanol (2 mL) was added to increase solubility and speed up the reactions. After stirring for an hour at room temperature, methanol was removed under reduced pressure and the products isolated as white solids. For analysis and further research application, the products were recrystallized using acetonitrile. If significant excess of **4** (more than 2%) was used or methanol was not completely removed the mixture dissolved fully in acetonitrile. The longer the alkyl chains

on cation and anion the better the yield due to the poorer solubility in acetonitrile and lower hygroscopicity (**5a** 70%, **5b** 90%, **5c** 80%, **5d** 40%, **5e** 60%, **5f** 15%). The attempt to make decyltrimethylammonium acetate was unsuccessful because of the complete solubility in acetonitrile.

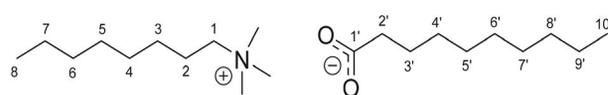
2. 3. Characterization of Alkyltrimethylammonium Carboxylates

N-Hexyl-*N,N,N*-trimethylammonium Decanoate (**5a**)



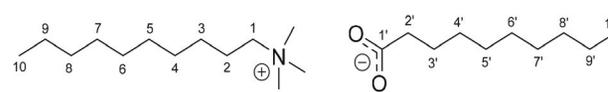
Prepared according to the general procedure B. White solid (11.5 g, 35%). IR 3473, 3393, 3020, 2920, 2852, 1572, 1377, 1055, 963, 643 cm⁻¹. ¹H NMR (500 MHz, CDCl₃) δ 3.42–3.35 (m, 2H, CH₂-1), 3.32 (s, 9H, NMe₃), 2.14–2.06 (m, 2H, CH₂-2'), 1.75–1.64 (m, 2H, CH₂-2), 1.59–1.48 (m, 2H, CH₂-3'), 1.38–1.15 (m, 18H, CH₂-chain), 0.92–0.78 (m, 6H, CH₃-10' and CH₃-6). ¹³C NMR (126 MHz, CDCl₃) δ 179.8 (COO), 66.9 (30.21), 53.2 (NMe₃), 39.4 (C-2'), 32.0, 31.4, 30.2, 29.9, 29.8, 29.50, 27.3, 26.0, 23.2, 22.8, 22.5, 14.2 (CH₃-6/CH₃-10'), 14.0 (CH₃-6/CH₃-10'). ¹⁵N NMR (51 MHz, CDCl₃) δ 49.5 (NMe₃). HRMS (ESI+): calcd. for C₉H₂₂N⁺ [M⁺] 144.1747, found 144.1749. HRMS (ESI-): calcd. for C₁₀H₁₉O₂⁻ [M⁻] 171.1391, found 171.1389.

N-Octyl-*N,N,N*-trimethylammonium Decanoate (**5b**)



Prepared according to the general procedure B. White solid (14.7 g, 63%). IR 3317, 3119, 3032, 2919, 2851, 1657, 1569, 1380, 972, 756 cm⁻¹. ¹H NMR (500 MHz, CDCl₃) δ 3.43–3.38 (m, 2H, CH₂-1), 3.38–3.31 (m, 9H, NMe₃), 2.19–2.05 (m, 2H, CH₂-2'), 1.76–1.61 (m, 2H, CH₂-2), 1.60–1.49 (m, 2H, CH₂-3'), 1.41–1.13 (m, 22H, CH₂-chain), 0.89–0.79 (m, 6H, CH₃-8 and CH₃-10). ¹³C NMR (126 MHz, CDCl₃) δ 179.6 (COO), 66.9 (C-1), 53.2 (NMe₃), 39.2 (C-2'), 32.0, 31.7, 30.2, 29.8, 29.8, 29.5, 29.3, 29.1, 27.3 (C-3'), 26.4, 23.2 (C-2), 22.8, 22.7, 14.2 (CH₃-8/CH₃-10'), 14.1 (CH₃-8/CH₃-10'). ¹⁵N NMR (51 MHz, CDCl₃) δ 49.7 (NMe₃). HRMS (ESI+): calcd. for C₁₁H₂₆N⁺ [M⁺] 172.2060, found 172.2057. HRMS (ESI-): calcd. for C₁₀H₁₉O₂⁻ [M⁻] 171.1391, found 171.1386.

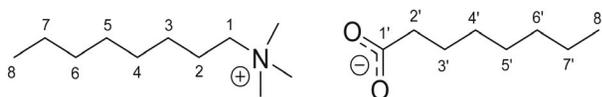
N-Decyl-*N,N,N*-trimethylammonium Decanoate (**5c**)



Prepared according to the general procedure B. White solid (25.3 g, 72%). IR 3429, 3348, 3195, 3119, 3032,

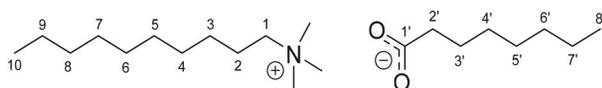
2916, 2850, 1656, 1566, 1387 cm^{-1} . ^1H NMR (500 MHz, CDCl_3) δ 3.42–3.35 (m, 2H, CH_2 -1), 3.32 (s, 9H, NMe_3), 2.16–2.07 (m, 2H, CH_2 -2'), 1.74–1.63 (m, 2H, CH_2 -2), 1.54 (p, $J = 7.2$ Hz, 2H, CH_2 -3'), 1.38–1.14 (m, 26H, CH_2 -chain), 0.91–0.79 (m, 6H, CH_3 -10 and CH_3 -10'). ^{13}C NMR (126 MHz, CDCl_3) δ 179.6 (COO), 66.9 (C-1), 53.2 (NMe_3), 39.0 (C-2'), 32.0, 31.9, 30.2, 29.9, 29.8, 29.54, 29.51, 29.4, 27.2 (C-3'), 26.4, 23.3 (C-2), 22.79, 22.75, 14.22 (CH_3 -10/ CH_3 -10'), 14.19 (CH_3 -10/ CH_3 -10'). ^{15}N NMR (51 MHz, CDCl_3) δ 49.5 (NMe_3). HRMS (ESI+): calcd. for $\text{C}_{13}\text{H}_{30}\text{N}^+$ [M^+] 200.2373, found 172.2373. HRMS (ESI-): calcd. for $\text{C}_{10}\text{H}_{19}\text{O}_2^-$ [M^-] 171.1391, found 171.1388.

N-Octyl-N,N,N-trimethylammonium Octanoate (5d)



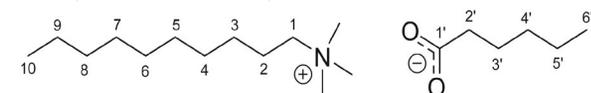
Prepared according to the general procedure B. White solid (10.8 g, 28%). IR 3510, 3022, 2919, 2851, 1657, 1573, 1378, 974, 918, 773 cm^{-1} . ^1H NMR (500 MHz, CDCl_3) δ 3.43–3.36 (m, 2H, CH_2 -1), 3.35–3.25 (m, 9H, NMe_3), 2.16–2.05 (m, 2H, CH_2 -2'), 1.74–1.61 (m, 2H, CH_2 -2), 1.59–1.46 (m, 2H, CH_2 -3'), 1.35–1.12 (m, 14H, CH_2 -chain), 0.89–0.75 (m, 6H, CH_3 -8 and CH_3 -8'). ^{13}C NMR (126 MHz, CDCl_3) δ 179.5 (COO), 66.8 (C-1), 53.1 (NMe_3), 39.1 (C-2'), 32.0, 31.7, 30.1, 29.5, 29.3, 29.1, 27.2 (C-3'), 26.3, 23.2 (C-2), 22.8, 22.6, 14.2 (CH_3 -8/ CH_3 -8'), 14.1 (CH_3 -8/ CH_3 -8'). ^{15}N NMR (51 MHz, CDCl_3) δ 49.4 (NMe_3). HRMS (ESI+): calcd. for $\text{C}_{11}\text{H}_{26}\text{N}^+$ [M^+] 172.2060, found 172.2054. HRMS (ESI-): calcd. for $\text{C}_8\text{H}_{15}\text{O}_2^-$ [M^-] 143.1078, found 143.1076.

N-Decyl-N,N,N-trimethylammonium Octanoate (5e)



Prepared according to the general procedure B. White solid (16.7 g, 54%). IR 3464, 3385, 3021, 2919, 2851, 1572, 1380, 915, 772, 722 cm^{-1} . ^1H NMR (500 MHz, CDCl_3) δ 3.45–3.39 (m, 2H, CH_2 -1), 3.40–3.35 (m, 9H, NMe_3), 2.23–2.11 (m, 2H, CH_2 -2'), 1.79–1.67 (m, 2H, CH_2 -2), 1.65–1.53 (m, 2H, CH_2 -3), 1.42–1.18 (m, 22H, CH_2 -chain), 0.93–0.81 (m, 6H, CH_3 -10 and CH_3 -8'). ^{13}C NMR (126 MHz, CDCl_3) δ 180.0 (COO), 67.1 (C-1), 53.4 (NMe_3), 39.3 (C-2'), 32.1, 32.0, 30.2, 29.6, 29.53, 29.49, 29.36, 29.34, 27.3 (C-3'), 26.4, 23.3 (C-2'), 22.9, 22.8, 14.3 (CH_3 -10/ CH_3 -8'), 14.2 (CH_3 -10/ CH_3 -8'). ^{15}N NMR (51 MHz, CDCl_3) δ 49.7 (NMe_3). HRMS (ESI+): calcd. for $\text{C}_{13}\text{H}_{30}\text{N}^+$ [M^+] 200.2373, found 172.2375. HRMS (ESI-): calcd. for $\text{C}_8\text{H}_{15}\text{O}_2^-$ [M^-] 143.1078, found 143.1073.

N-Decyl-N,N,N-trimethylammonium Hexanoate (5f)



Prepared according to the general procedure B. White solid (2.2 g, 13%). IR 3359, 3021, 2922, 2854, 1572, 1377, 973, 918, 766, 644 cm^{-1} . ^1H NMR (500 MHz, CDCl_3) δ 3.45–3.37 (m, 2H, CH_2 -1), 3.38–3.30 (m, 9H, NMe_3), 2.21–2.03 (m, 2H, CH_2 -2'), 1.76–1.61 (m, 2H, CH_2 -2), 1.61–1.50 (m, 2H, CH_2 -3'), 1.38–1.15 (m, 18H, CH_2 -chain), 0.92–0.78 (m, 6H, CH_2 -8 and CH_2 -). ^{13}C NMR (126 MHz, CDCl_3) δ 179.6 (COO), 66.9 (C-1), 53.2 (NMe_3), 39.2 (C-2'), 32.4, 31.9, 29.50, 29.46, 29.3, 26.9 (3'), 26.4, 23.3 (C-2), 22.8, 22.7, 14.3 (CH_3 -10/ CH_3 -6'), 14.2 (CH_3 -10/ CH_3 -6'). ^{15}N NMR (51 MHz, CDCl_3) δ 49.8 (NMe_3). HRMS (ESI+): calcd. for $\text{C}_{13}\text{H}_{30}\text{N}^+$ [M^+] 200.2373, found 200.2370. HRMS (ESI-): calcd. for $\text{C}_{10}\text{H}_{19}\text{O}_2^-$ [M^-] 171.1391, found 171.1386.

3. Results and Discussion

A series of quaternary ammonium carboxylates – *N*-alkyl-*N,N,N*-trimethylammonium alkanecarboxylates were prepared according to two general procedures as presented on Figure 2.

In the first step of procedure A, where the reaction mixture was heated at 80 °C for 3 days, alkyltrimethylammonium chlorides (alkyl = hexyl, **3a**; octyl, **3b**; decyl, **3c**) were obtained. Procedure A is followed by a cost effective approach to anion exchange in ethanolic solutions of KOH through precipitation of KCl (see Experimental Section for details) yielding alkyltrimethylammonium alkanecarboxylates (**5a–e**, Table in Figure 2). Even though the reported solubility of KCl in ethanol is very low (0.034%)¹⁴ the final products of procedure A shown in Table 1 contained up to 3% of inorganic salt as determined by AgNO_3 titration ($0.6 \pm 0.5\%$ Cl^-) and TG analysis ($2 \pm 1\%$). Presumably the presence of the product significantly increases solubility of KCl in ethanol. In fact, products are soluble in polar (water, methanol, ethanol, 1-butanol, acetone, ethyl acetate) and most non-polar (diethyl ether, *n*-hexane, toluene, dichloromethane, chloroform) solvents, making complete separation from KCl by precipitation impossible.

Therefore, the procedure B was developed as an alternative. Starting from dimethylamine (**6**) the long alkyl chain is introduced in the step before quaternization. Therefore, the soluble halide salts are easily washed away with water. Quaternization was successfully achieved with dimethyl carbonate (Me_2CO_3), a common methylating agent enabling a facile purification method. The side products, methanol and CO_2 , are easily removed under reduced pressure. Jiang *et al.* used significant excess (5–10 eq) of Me_2CO_3 and relatively short reaction times (5–7 h).^{8,9} We extended the reaction time in attempt to reach quantitative yield with minimal excess of reactant. Anion exchange was performed with carboxylic acid followed by the removal of volatile byproducts. For analysis the products (**5a–f**) were recrystallized from acetonitrile. Surfactants with longer alkyl chains to the ones in this work have been reportedly

For comparison TG/DSC curve of decyltrimethylammonium chloride (**3c**) is also presented on Figures S1 and S2 indicating lower thermal stability of carboxylates. Stability is also somewhat lowered by KCl (Table 1). Melting point or phase transition was observed for some of the products. KCl lowers the temperature of this transition.

4. Conclusions

In the proposed salt-free procedure first alkyl trimethylammonium methylcarbonate is obtained and reacted with carboxylic acid. The side products of methanol and CO₂ are easily removed under reduced pressure. In principle, salts composed of quaternary ammonium cations and most anions can be synthesized with this procedure. Reactants are inexpensive thus the procedure is potentially applicable in industrial production. An alternative procedure applying precipitation method is a viable alternative when inorganic impurities do not affect the application of the surfactant.

Supplementary Material

Copies of the IR and NMR spectra as well as additional figures on thermal analysis are available free of charge.

5. Acknowledgements

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Povzetek

Površinsko aktivne snovi v komercialnih produktih pogosto vsebujejo mešanice anionskih in kationskih surfaktantov, zato so bile dosedaj večinoma preiskovane vodne raztopine mešanic površinsko aktivnih snovi. Vendar pa so raziskave čistih katanionskih površinsko aktivnih snovi, ki se pogosto imenujejo katanionski surfaktanti brez soli, redke. Ena od težav je pridobivanje vzorcev z zahtevano čistostjo zaradi težkega ločevanja teh spojin od anorganskih soli. V tem delu predstavljamo alternativno metodo sinteze z dimetilkarbonatom kot alkilnim sredstvom za pripravo alkil trimetilamonijevih alkankarboksilatov s srednjimi dolžinami alkilnih verig (6–10).



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