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The surface properties of amine oxides with a fluoroether chain

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ABSTRACT

Persistent organic pollutants (POPs) includes long-chained fluorosurfactants, for instance, perfluorooctanonate (PFOA) and perfluorooctanesulfonate (PFOS). In order to find out alternative fluorosurfactants, we synthesized eight amine oxides with a fluoroether chain. Their surface properties were evaluated and compared with perfluoroalkyl and hydrocarbon analogues. The surface tensions at critical micelle concentration (γ_{cmc}) for the eight amine oxides with a fluoroether chain were at a range from 15.5 to 23.0 mN/m, and γ_{cmc} of four fluoroether amine oxides were below 17 mN/m comparable to perfluorooctyl analogues (16.4 mN/m) and much lower than perfluorohexyl (20.5 mN/m) and hydrocarbon analogues (24.2 and 24.7 mN/m). The critical micelle concentration (cmc) for the eight amine oxides with a fluoroether chain were 3 to 535 $\times$ 10^{-4} mol/L. The cmc of four fluoroether amine oxides were 3 to 21 $\times$ 10^{-4} mol/L (0.2 to 1.0 g/L) comparable to perfluorooctyl (0.4 g/L) and hydrocarbon analogues (4 and 10 $\times$ 10^{-4} mol/L) and much lower than perfluorohexyl analogue (36.9 g/L). The surface excesses, the limiting molecule areas and the free energies of micellization of amine oxides were calculated. Fluoroether surfactants are promising alternatives for PFOA and PFOS.

1. Introduction

Amine oxides are amphoteric surfactants, showing nonionic characteristics in neutral or alkaline solutions, and cationic characteristics in acidic solutions. Amine oxide surfactants have good foaming properties, wettability, thickening, low toxic and biodegradability, and they cause less irritation to the skin [1]. Because of these properties, amine oxide surfactants are widely used in detergents, shampoos, cosmetics and textile auxiliaries. Amine oxide surfactants are also used as foam boosters and stabilizers. On the other hand, fluorinated surfactants usually have high thermal and chemical stability. Furthermore, fluorinated surfactants have higher surface activities at lower critical micelle concentrations (cmc) compared to the traditional surfactants. Therefore, fluorinated surfactants play a special role in many applications such as electroplating, fire-fighting foams and repellents [2].

However, according to the Stockholm Convention, long-chained perfluoroalkyl substances such as perfluorooctanonate (PFOA) and perfluorooctanesulfonate (PFOS) are classified as organic persistent pollutants (POPs) because they are toxic, persistent, bioaccumulative and able to transfer to long distance in environment [3]. Although shortening fluorinated segments were proposed to reduce toxicity, the cmc and the surface tension (γ) will increase with the shortening of the chain. Fluorosurfactants with high surface activities and environmentally friendly properties are in a high demand in order to replace the compounds with long-chained perfluoroalkyl groups. We have been focusing on the risk evaluation of several kinds of emerging fluorosurfactants including their occurrence in environment and livings and also their toxicities to aquatic organisms and mammals [4–8]. Assisted by the biological and environmental research, we designed gemini cationic surfactants with flexible perfluorinated-ether chains last year [9]. We wish that newly-designed structures with a fluoroether chain bring more understanding about structural effect on not only surface property but also bioaccumulative ability. While the alkyl amine oxide surfactants are well researched,-very few research on fluorinated amine oxides has been reported [10]. The first fluoroether amine oxide dates back to 50 years ago [10e]. Commercially available fluoroether

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surfactants, such as GenX and ADONA, are anionic surfactants using in fluoropolymer manufacture. Amerduri et al. have developed CH_2-containing alternatives based on PVDF [10f,g]. Comparative structure-property correlations for amine-oxide surfactants with fluoroether and fluoroalkyl tail structures are needed.

In this article, we report our recent study on perfluoroether amine oxides and their performance together with a comparison with perfluoroalkyl amine oxides and aliphatic amine oxides.

2. Result and discussion

We synthesized tertiary amines from fluorinated esters, carbonyl fluorides, carbonyl chlorides or acids. The tertiary amines were oxidized to provide amino oxides for surface properties studies. (Scheme 1)

After we obtained the amine oxides surfactants, the surface tensions in gradient concentration were measured. The surface tensions at cmc (γ_{cmc}), cmc, the surface excesses (Γ_{max}), the limiting molecule areas (A_{min}) and the free energies of micellization (ΔG^o) of amine oxides were calculated and depicted in Table 1.

The surface excess Γ_{max} (also written as Γ_{cmc}) and the limiting molecule area A_{min} (also written as A_{cmc}) were used to characterize the behavior of surfactant molecules at air-water interface. Γ_{max} is defined as the concentration of surfactant molecules in a surface plane relative to that at a similar plane in the bulk at cmc. A_{min} corresponds to the area per surfactant molecule at air-water interface at cmc [2]. We calculated Γ_{max} and A_{min} by using the Gibbs equation

$$\Gamma_{max} = -\frac{1}{2.303 \times nRT} \times \frac{d\gamma}{d\log c}$$

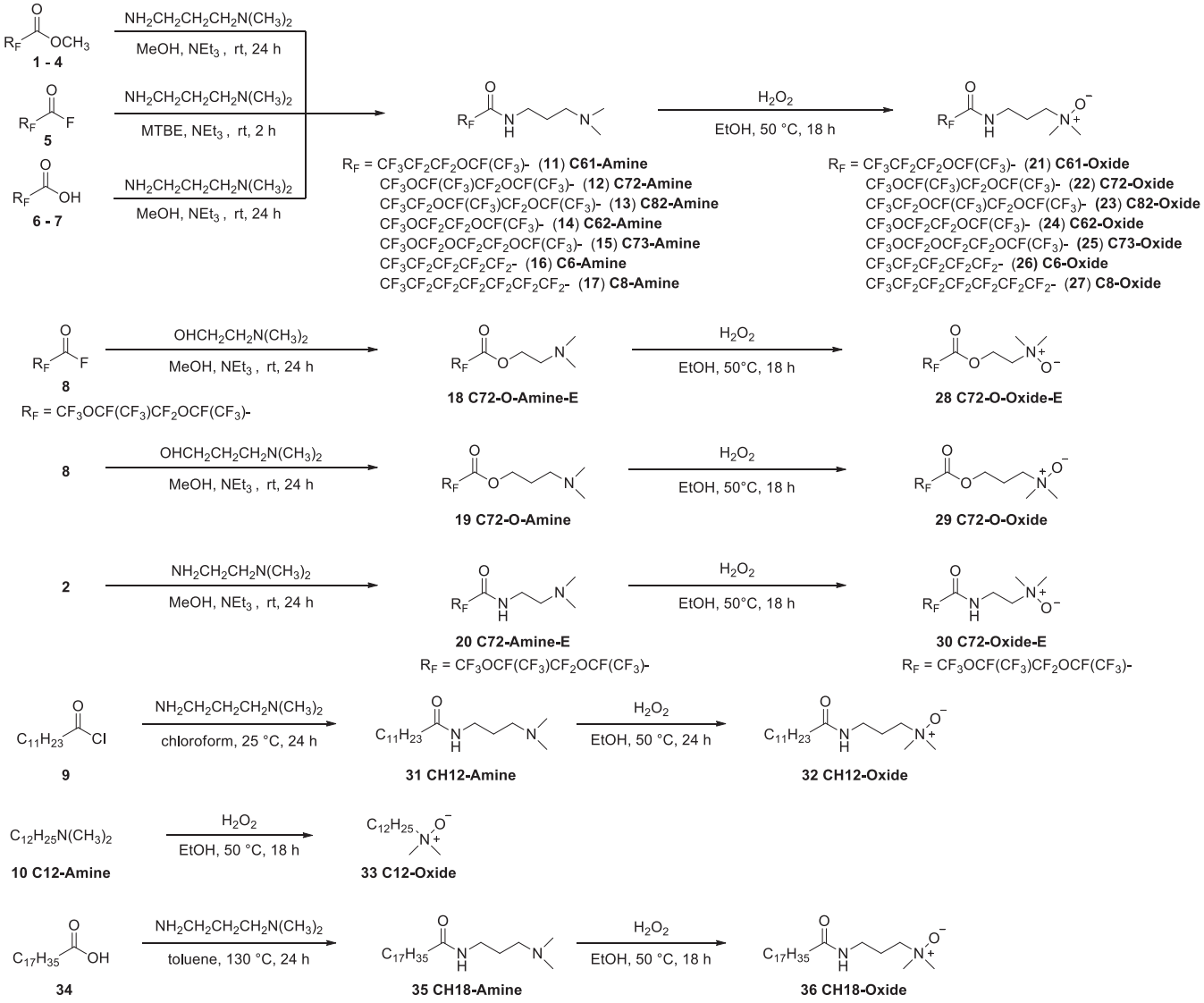
and the equation

$$A_{min} = \frac{10^{14}}{N_A \Gamma_{max}}$$

The "n" was taken as 1 for amine oxide surfactants because they were looked as a nondissociating molecule in a neutral aqueous solution.

The free energies of micellization (ΔG^o) were calculated based on the equation $\Delta G^o = RT\ln$cmc.

In the Table 1 and Fig. 1, the γ_{cmc} of compounds were in an order as **C61-Oxide 21** > **C62-Oxide 24** > **C72-Oxide 22** > **C73-Oxide 25** > **C82-Oxide 23**. The same order was found with cmc of these five surfactants. **C61-Oxide 21** has been reported with a surface tension of 28.5 mN/m in 1% aqueous solution [10e], which is higher than our result (18.4 mN/m). The surface tensions and cmc were determined by hydrophobic fluorinated chains if hydrophilic groups were same. The number of fluorinated carbons was prominent on the reducing of γ and



Scheme 1. The synthesis of amine oxides with fluoroether, perfluoroalkyl and aliphatic groups.

Table 1
The surface properties of amine oxides.

Compound		γ_{cmc} (mN/m)	cmc ($\times 10^{-4}$ mol/L)	(g/L)	Γ_{max} ($\times 10^{-10}$ mol/cm^2)	A_{min} (nm^2)	ΔG^o (kJ/mol)	Purity[a]	T (^{o}C)
21	**C61-Oxide**	18.4	159	6.9	2.8	0.59	-10	98%	23
22	**C72-Oxide**	16.8	21	1.0	3.3	0.51	-15	96%	22
23	**C82-Oxide**	15.5	3	0.2	2.8	0.60	-20	92%	22
24	**C62-Oxide**	17.0	88	3.9	3.3	0.50	-12	96%	23
25	**C73-Oxide**	15.8	14	0.7	0.8	2.15	-16	98%	22
26	**C6-Oxide**	20.5	891	36.9	2.4	0.68	-6	96%	24
27	**C8-Oxide**	16.4	7	0.4	2.8	0.59	-18	96%	24
28	**C72-O-Oxide-E**	23.0	472	22.8	2.5	0.68	-8	94%	23
29	**C72-O-Oxide**	21.4	535	26.6	1.0	1.63	-7	98%	22
30	**C72-Oxide-E**	16.6	19	0.9	2.8	0.59	-15	99%	24
32	**CH12-Oxide**	24.2	4	0.1	3.4	0.50	-19	95%	22
33	**C12-Oxide**	24.7	10	0.2	2.4	0.69	-17	99%	22

[a] The purity were analyzed by the NMR spectroscopy (see supplementary material)

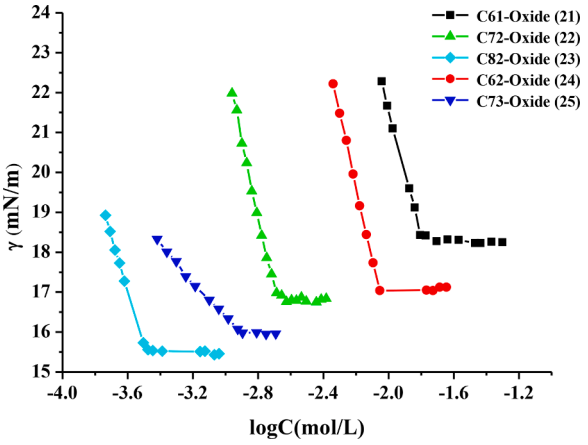


Fig. 1. The γ-logC curves for fluoroether amine oxides.

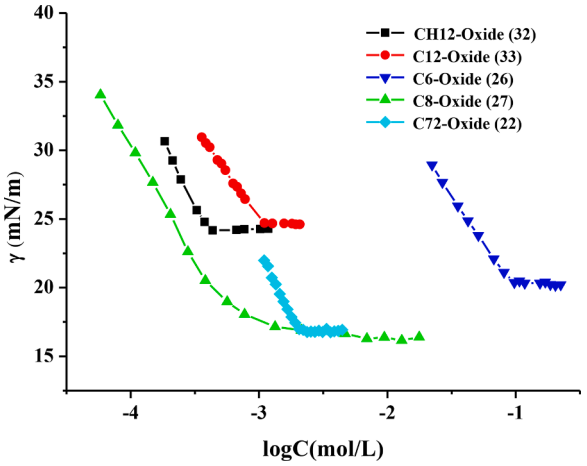


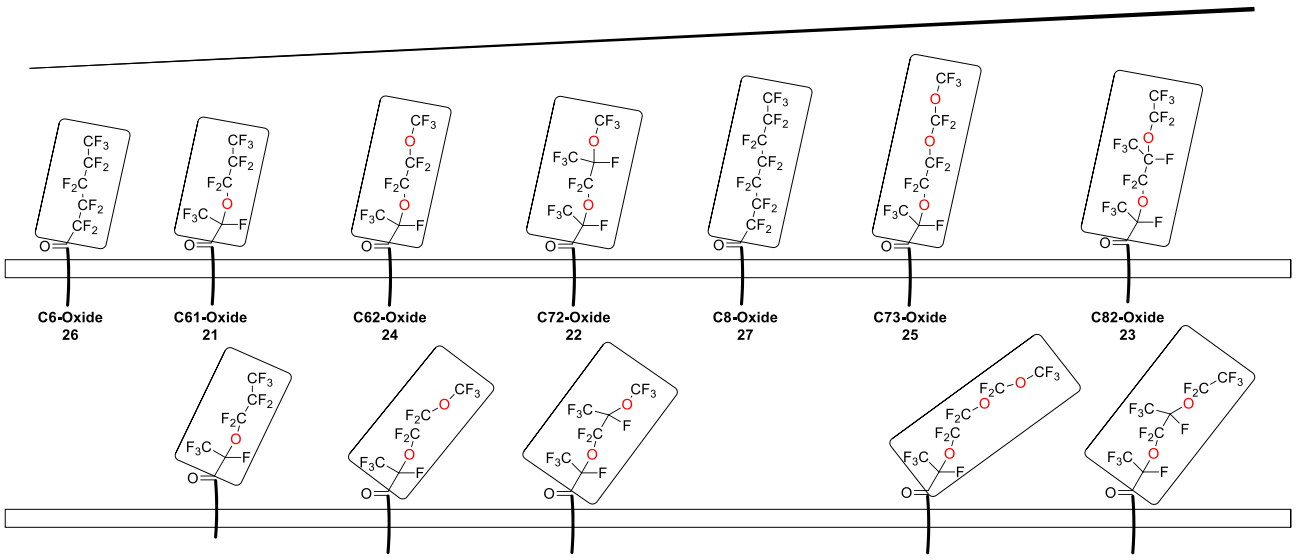
Fig. 2. The γ-logC curves for fluoroether amine oxide **22** compared with perfluoroalkyl and aliphatic amine oxides.

cmc. Increasing oxygen number on the hydrophobic fluorinated segments reduced γ and cmc as well. Introducing oxygen atoms into fluorinated chains increased the hydrophobic properties of fluorosurfactants [11]. Γ_{max} and A_{min} were calculated by the slope of the γ-logC curves. The slope of **C73-Oxide 25** was smaller than others and its Γ_{max} was smallest and A_{min} was largest. This could be explained by the high flexibility of the oxygen-rich hydrophobic fluorinated chain within **C73-Oxide 25** molecule [9]. The Γ_{max} and A_{min} of the other four surfactants (**C61-Oxide 21, C62-Oxide 24, C72-Oxide 22** and **C82-Oxide 23**) were very close. The trend ofΔG^o was based on the trend of cmc. The order ofΔG^o was the same as that for cmc.

In the Table 1 and Fig. 2, we compared fluoroether **C72-Oxide 22** with fluorinated amine oxide surfactants (**C6-Oxide 26** and **C8-Oxide 27**) and aliphatic amine oxide surfactants (**CH12-Oxide 32** and **C12-Oxide 33**). The surface properties of **C72-Oxide 22** and **C8-Oxide 27** were close. The γ_{cmc} for **C72-Oxide 22** and **C8-Oxide 27** were 16.8 and 16.4 mN/m and the cmc for **C72-Oxide 22** and **C8-Oxide 27** were 21 and 7 $\times 10^{-4}$ mol/L (1.0 and 0.4 g/L), respectively. Surface tension was determined majorly by the fluorinated segment. The perfluoroalkyl chain $CF_3(CF_2)_6$- behaved comparable to the fluoroether chain $CF_3OCF(CF_3)CF_2OCF(CF_3)$-. One fluorinated carbon worked roughly as two oxygen atoms within the fluoroether chain. Shortening two carbons in the perfluoroalkyl chain gave **C6-Oxide 26**, resulting a sharp increase of γ_{cmc} (20.5 mN/m) and cmc (36.9 g/L). Although the γ_{cmc} of $CF_3(CF_2)_4$-amine oxide **26** was lower than those of the two hydrocarbon surfactants **CH12-Oxide 32** and **C12-Oxide 33**, the cmc of aliphatic **CH12-Oxide 32** and **C12-Oxide 33** were closer to those of fluorinated **C72-Oxide 22** and **C8-Oxide 27** probably due to the similar hydrophilic-lipophilic balance (HLB) and lower than that of **C6-Oxide 26**. Increasing carbon chain length in compounds **36 CH18-oxide** with a $C_{17}H_{35}$ chain resulted in a extremely low solubility and failed to give a low surface tension. The γ of hydrocarbon **32** and **33** were higher than 24 mN/m, much higher than that of fluorinated surfactant. The low polarizability and weak dispersion force for perfluorinated carbon and ether chains with an enough length can reduce not only surface tension but also cmc [2,12]. The cmc relates to the efficiency and the surface tension relates to effectiveness [2]. Only fluorinated surfactants can satisfy both requirements to reach low surface tension and low cmc.

An illustration in Scheme 2 may help to understand the air-water interfacial behavior of the surfactants bearing with various fluorinated chains. The surface tensions were determined majorly by the length of perfluoroalkyl or perfluoroether chains. When we discuss the term "length" here, both backbond and branched chains were considered. As we discussed above, the effect of two ether bonds on surface tension will be vaguely equal to that of a difluoromethylene group. So, the surface tensions were consistent with the apparent lengths when the surfactant molecules were packing tightly in the air-water interfaces.

In Scheme 2, the flexibility of fluorinated chains resulting from the ether bonds were illustrated. As we all know, silicone surfactants enhance their surface activities by the multiple flexible ether bonds [2b]. Fluoroether chains can enhance the surface activities by the same mechanism. The most extraordinary example is **C73-Oxide 25** with the limiting molecular area of 2.15 nm^2, much larger than others. We contributed the wide A_{min} of **C73-Oxide 25** to the three flexible ether bonds. While flexibility has an essential influence on A_{min}, the bulk of fluorinated groups change the area covered by surfactant molecules when they are saturated and packing tightly in the interface. The A_{min} of **C62-Oxide 24, C72-Oxide 22, C8-Oxide 27** and **C82-Oxide 23** were



Scheme 2. An illustration to explain the effect of perfluoroalkyl and fluoroether chain on the surface tension and the limiting molecular area.

reasonable according to this explanation. The large limiting molecular areas of **C61-Oxide 21** and **C6-Oxide 26** may be attributed to the lower surface excess Γ_{max} for higher solubilities of short-chained fluorinated surfactants in water.

In the Table 1 and Fig. 3, we examined linkage effect by comparing fluoroether **C72-Oxide 22** with ester linkage amine oxides **C72-O-Oxide 29** and **C72-O-Oxide-E 28** and shorter linkage amine oxides (**C72-Oxide-E 30**). Ester linkages increased cmc and γ_{cmc} dramatically. By shortening propylene (-$CH_2CH_2CH_2$-) to ethylene (-CH_2CH_2-), slightly change of **C72-Oxide-E 30** in γ_{cmc} (16.6 mN/m) and cmc (0.9 g/L) were found. Due to the lone-pair electron resonance of nitrogen atom and carbonyl group, amide bonds are normally rigid compared with ester bonds [12]. The surfactants with amide bonds are less flexible and consequently have lower entropies compared with the surfactants with ester bonds, and tend to be pushed out of the bulk aqueous environment to the air-water interface. Thus, the surface excess of **C72-Oxide 22** was larger than that of **C72-O-Oxide 29**, and the limiting molecular area of **C72-Oxide 22** was smaller than that of **C72-O-Oxide 29** (Table 1). We also observed higher solubility of **C72-O-Oxide 29** and **C72-O-Oxide-E 28** than that of **C72-Oxide 22** and **C72-Oxide-E 30**. The high surface tension and cmc of ester surfactants **28** and **29** could also be attributed to their solubilities of monomers inside the bulk aqueous solution.

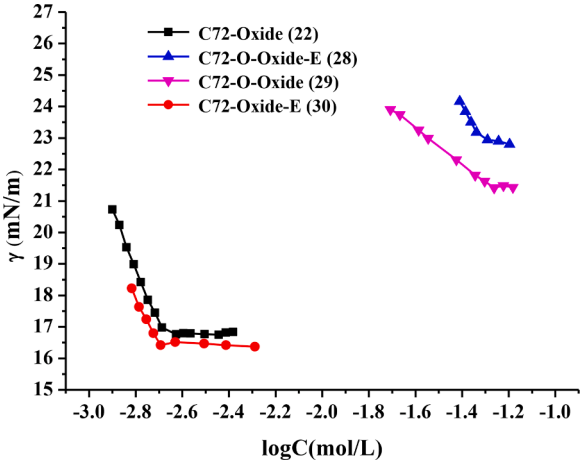


Fig. 3. The γ-logC curves for amine oxides with different alkyl chain lengths and different linkage groups.

3. Conclusion

We have synthesized thirteen surfactants including fluoroether, perfluoroalkyl and aliphatic amine oxides. Their surface properties were examined and compared. The γ_{cmc} and cmc of fluoroether surfactants can reach as low as 15.5 mN/m and 0.2 g/L. By adjusting oxygen and carbon number of fluoroether chains, we can provide surfactants with properties comparable to PFOA-based amino oxides. Oxygen atoms within fluoroether chain, which can reduce surface tension and cmc, are hydrophobic. Fluorinated surfactants are irreplaceable for their properties of both low surface tension and cmc. Modification of surfactants by fluoroether chains provide a alternative strategy for fluorinated POPs.

4. Experimental section

4.1. General information

Fluorinated starting materials were provided by Sanming Hexafluo Chemicals Co., LTD. (see SI), and all other reagents were of AR grade quality and used without further purification. Chemical shifts are expressed in parts per million (ppm) with respect to the residual solvent peak. Chemical shifts for ^{19}F NMR are reported in ppm downfield from fluorotrichloromethane ($CFCl_3$). ^{13}C NMR was broad-band decoupled from hydrogen nuclei. Coupling constants are reported as hertz (Hz). Signal shapes and splitting patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. The resonances corresponding the N-H protons are not included in the analysis – possibly due to exchange broadening. HRMS (ESI) data were tested on a Water Micromass GCT Premier. Surface tensions were obtained with KRÜSS(DLSB-5L/-20) surface tension meter by Wilhelmy platinum plate method. Specific test information is recorded in supplementary material.

4.2. Experimental procedures and characterization data for compounds

N-(3-(dimethylamino)propyl)-2,3,3,3-tetrafluoro-2-(perfluoropropoxy)propanamide (**11**) **C61-Amine** [9]

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added 3-dimethylaminopropylamine (20 mmol, 2.04 g, 1 eq.), triethylamine (30 mmol, 3.04 g, 1.5 eq.) and methanol (10 mL). Then, compound $CF_3CF_2CF_2OCF(CF_3)COOCH_3$ (**1**, 20 mmol, 6.88 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. After the reaction the solvent was removed by rotary

evaporation under vacuum. Dichloromethane (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product was a light-yellow liquid (8.04 g, 97%) [9]. ^{1}H NMR (400 MHz, CD_3OD) δ 3.37 – 3.27 (m, 2H), 2.33 (t, J = 7.6 Hz, 2H), 2.20 (s, 6H), 1.75 – 1.67 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -81.9 (dm, J = 145.9 Hz, 1F), -82.9 (t, J = 7.1 Hz, 3F), -84.6 (d, J = 1.9 Hz, 3F), -86.2 (dm, J = 149.3 Hz 1F), -131.2 (s, 2F), -134.0 (dd, J = 19.6, 6.8 Hz, 1F). IR (film) ν/cm^{-1}: 3342.1, 2954.7, 2869.8, 2830.6, 2790.3, 1715.9, 1533.1, 1470.5, 1343.1, 1322.2, 1296.4, 1235.4, 1200.1, 1163.3, 1133.1, 1107.8, 1062.5, 1041.6, 991.0, 809.1, 747.3, 719.8, 628.6, 534.8.

N-(3-(dimethylamino)propyl)-2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(trifluoromethoxy)propoxy)propanamide (**12**) **C72-Amine**

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added 3-dimethylaminopropylamine (20 mmol, 2.04 g, 1 eq.), triethylamine (30 mmol, 3.04 g, 1.5 eq.) and methanol (10 mL). Then, compound $CF_3OCF(CF_3)CF_2OCF(CF_3)COOCH_3$ (**2**, 20 mmol, 8.20 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. After the reaction the solvent was removed by rotary evaporation under vacuum. Dichloromethane (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product was a light-yellow liquid (8.84 g, 92%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.38 – 3.30 (m, 2H), 2.34 (t, J = 7.6 Hz, 2H), 2.22 (s, 6H), 1.77 – 1.69 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -55.0 (m, 3F), -80.8 (m, 1F), -81.5 (m, 3F), -84.1 (dd, J = 6.4, 1.5 Hz, 3F), -84.5 (m, 1F), -133.9 (m, 1F), -147.8 (m, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 157.8 (m), 56.6, 43.9, 38.1, 25.9, carbons corresponding to the $CF_3OCF(CF3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{12}H_{14}F_{13}N_2O_3$ $[M+H]^+$: 481.0791 found: 481.0793. IR (film) ν/cm^{-1}: 3342.6, 2955.0, 2869.7, 2830.6, 2790.8, 1724.6, 1533.7, 1471.0, 1239.0, 1161.4, 1106.3, 1077.6, 1062.6, 1041.8, 982.1, 892.7, 809.2, 765.6, 739.1, 683.9, 643.8, 534.3.

N-(3-(dimethylamino)propyl)-2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(perfluoroethoxy)propoxy)propanamide (**13**) **C82-Amine**

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added 3-dimethylaminopropylamine (20 mmol, 2.04 g, 1 eq.), triethylamine (30 mmol, 3.04 g, 1.5 eq.) and methanol (10 mL). Then, compound $CF_3CF_2OCF(CF_3)CF_2OCF(CF_3)COOCH_3$ (**3**, 20 mmol, 9.20 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. After the reaction the solvent was removed by rotary evaporation under vacuum. Dichloromethane (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product was obtained as a light-yellow liquid (9.76 g, 92%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.38 – 3.32 (m, 2H), 2.34 (t, J = 7.4 Hz, 2H), 2.22 (s, 6H), 1.77 – 1.69 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -81.3 (m, 3F), -84.2 (m, 3F), -80.7 – -84.7 (m, 2F), -87.0 (m, 2F), -88.3 (d, J = 15.8 Hz, 3F), -133.8 (dd, J = 19.6, 7.1 Hz, 1F), -146.4 (t, J = 22.0 Hz, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 157.8 (d, J = 26 Hz), 56.6, 43.9, 38.1, 25.9, carbons corresponding to the $CF_3CF_2OCF(CF_3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{13}H_{14}F_{15}N_2O_3$ $[M+H]^+$: 531.0759, found: 531.0754. IR (film) ν/cm^{-1}: 3342.8, 2955.5, 2870.6, 2831.5, 2790.8, 1716.5, 1533.7, 1470.9, 1305.5, 1235.2, 1153.5, 1125.3, 1090.3, 1041.7, 981.8, 916.4, 825.6, 805.8, 765.2, 743.5, 720.6, 691.2, 646.0, 523.5.

N-(3-(dimethylamino)propyl)-2,3,3,3-tetrafluoro-2-(1,1,2,2-tetrafluoro-2-(trifluoromethoxy)ethoxy)propanamide (**14**) **C62-Amine** [9]

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added 3-dimethylaminopropylamine (20 mmol, 2.04 g, 1 eq.), triethylamine (30 mmol, 3.04 g, 1.5 eq.) and methanol (10 mL). Then, compound $CF_3OCF_2CF_2OCF(CF_3)COOCH_3$ (**4**, 20 mmol, 7.20 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. After the reaction the solvent was removed by rotary evaporation under vacuum. Dichloromethane (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product was obtained as a light-yellow liquid (8.26 g, 96%).[9] This compound was obtained as a light-yellow liquid. ^{1}H NMR (400 MHz, CD_3OD) δ 3.34 – 3.30 (m, 2H), 2.33 (t, J = 7.6 Hz, 2H), 2.21 (s, 6H), 1.75 – 1.68 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -57.0 (t, J = 9.0 Hz, 3F), -84.1 (d, J = 1.9 Hz, 3F), -86.2 (dd, J = 146.6, 18.4 Hz, 1F), -90.3 (dd, J = 146.6, 8.3 Hz, 1F), -91.9 (m, 2F), -134.1 (dd, J = 18.1, 7.9 Hz, 1F). IR (film) ν/cm^{-1}: 3342.5, 2954.7, 2869.9, 2830.6, 2790.3, 1723.5, 1533.6, 1470.4, 1285.2, 1224.0, 1149.8, 1114.1, 1061.8, 1041.5, 988.2, 902.2, 795.8, 765.5, 720.7, 681.3, 616.5.

2-(2-(difluoro(trifluoromethoxy)methoxy)-1,1,2,2-tetrafluoroethoxy)-N-(3-(dimethylamino)propyl)-2,3,3,3-tetrafluoropropanamide (**15**) **C73-Amine** [9]

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added 3-dimethylaminopropylamine (20 mmol, 2.04 g, 1 eq.), triethylamine (30 mmol, 3.04 g, 1.5 eq.) and *tert*-butyl methyl ether (10 mL). Then, compound $CF_3OCF_2OCF_2CF_2OCF(CF_3)COF$ (**5**, 20 mmol, 8.28 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 4 h. After the reaction the solvent was removed by rotary evaporation under vacuum. Dichloromethane (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product was obtained as a light-yellow liquid (9.71 g, 94%) [9]. ^{1}H NMR (400 MHz, CD_3OD) δ 3.40 – 3.32 (m, 2H), 2.42 (t, J = 7.6 Hz, 2H), 2.28 (s, 6H), 1.82 – 1.75 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -55.3 (m, 2F), -58.7 (t, J = 8.8 Hz, 3F), -84.1 (d, J = 1.5 Hz, 3F), -86.7 (m, 1F), -90.1 (m, 1F), -91.7 (q, J = 21.1, 10.5 Hz, 2F) -134.0 (dd, J = 18.4, 7.9 Hz, 1F). IR (film) ν/cm^{-1}: 3336.3, 2956.5, 2871.2, 2832.1, 1714.9, 1534.2, 1471.0, 1390.5, 1309.5, 1219.9, 1108.1, 1040.7, 989.3, 964.1, 869.4, 794.7, 765.3, 720.6, 647.1.

N-(3-(dimethylamino)propyl)-2,2,3,3,4,4,5,5,6,6,6-undecafluorohexanamide (**16**) **C6-Amine** [13]

3-dimethylaminopropylamine (20 mmol, 2.04 g, 1 eq.), triethylamine (30 mmol, 3.04 g, 1.5 eq.) and methanol (10 mL) was added into a 100 mL three necked round-bottom flask. The mixture was stirred evenly at room temperature. Then, compound $CF_3CF_2CF_2CF_2COOH$ (**6**, 20 mmol, 6.28 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. The solvent was removed by rotary evaporation under vacuum. The final product was obtained as a light-yellow liquid (7.65 g, 96%). ^{1}H NMR (400 MHz, CD_3OD) δ 2.92 (t, J = 6.8 Hz, 2H), 2.44 (t, J = 7.2 Hz, 2H), 2.24 (s, 6H), 1.79 – 1.72 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -82.5 (m, 3F), -118.0 (m, 2F), -123.8 (m, 4F), -127.5 (m, 2F). IR (film) ν/cm^{-1}: 3429.6, 2959.5, 2835.0, 1685.8, 1471.6, 1394.6, 1350.1, 1237.8, 1204.7, 1146.6, 1106.8, 1083.0, 867.9, 827.5, 807.9, 747.6, 732.5, 713.8, 655.7, 566.3, 530.4.

N-(3-(dimethylamino)propyl)-2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluorooctanamide (**17**) **C8-Amine** [14]

3-dimethylaminopropylamine (20 mmol, 2.04 g, 1 eq.), triethylamine (30 mmol, 3.04 g, 1.5 eq.) and methanol (10 mL) was added into 100 ml three necked round-bottom flask. Stir the mixture evenly at room temperature. Then, compound $CF_3(CF_2)_6COOH$ (**7**, 20 mmol, 8.28 g, 1.0 eq.) was slowly dropped in and the mixture were stirred at room temperature for 24 h. The solvent was removed by rotary evaporation under vacuum. The final product was obtained as a white solid (8.99 g, 94%). ^{1}H NMR (400 MHz, CD_3OD) δ 2.90 (t, J = 7.2 Hz, 2H), 2.42 (t, J = 7.2

Hz, 2H), 2.22 (s, 6H), 1.74 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -82.5 (m, 3F), -118.0 (m, 2F), -122.7 (m, 2F), -123.1 (m, 2F), -123.6 (m, 2F), -123.9 (m, 2F), -127.4 (m, 2F). IR (film) ν/cm^{-1}: 2960.6, 2836.1, 1686.1, 1472.9, 1394.2, 1360.4, 1240.9, 1206.3, 1149.0, 1102.1, 1013.1, 812.9, 746.6, 721.9, 700.0, 663.8, 641.5, 560.8, 530.4.

2-(dimethylamino)ethyl2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(trifluoromethoxy)propoxy)propanoate (**18**) **C72-O-Amine-E**

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added 2-(dimethylamino)ethan-1-ol (80 mmol, 7.13 g, 4 eq.), methanol (10 mL). Then, compound $CF_3OCF(CF_3)CF_2OCF(CF_3)$ COF (**8**, 20 mmol, 7.96 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. After the reaction the solvent was removed by rotary evaporation under vacuum. Ethyl acetate (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product was obtained as a white viscous solid (8.04 g, 86%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.67 (t, J = 5.6 Hz, 2H), 2.57 (t, J = 6.0 Hz, 2H), 2.34 (s, 6H). ^{19}F NMR (376 MHz, CD_3OD) δ -54.9 (m, 3F), -81.8 (m, 3F), -80.4 – -84.9 (m, 2F), -83.9 (m, 3F), -126.5 (m, 1F), -147.8 (m, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 162.4 (m), 61.9, 59.6, 45.4, carbons corresponding to the $CF_3OCF(CF_3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-EI (m/z) calcd for $C_{11}H_{10}F_{13}NO_4$ $[M-H]^-$: 467.0397, found: 467.0393. IR (film) ν/cm^{-1}: 3675.9, 3412.4, 2839.4, 1697.4, 1464.5, 1403.7, 1355.8, 1232.3, 1157.9, 1040.3, 980.7, 892.7, 820.1, 771.4, 738.9, 682.9, 653.0, 619.6, 537.3.

2-(dimethylamino)propyl2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(trifluoromethoxy)propoxy)propanoate (**19**) **C72-O-Amine**

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added 3-(dimethylamino)propan-1-ol (100 mmol, 10.3 g, 4 eq.), methanol (10 mL). Then, compound $CF_3OCF(CF_3)CF_2OCF(CF_3)$ COF (**8**, 25 mmol, 9.95 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. After the reaction the solvent was removed by rotary evaporation under vacuum. Ethyl acetate (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product was obtained as a white viscous solid (10.11 g, 84%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.57 (t, J = 6.4 Hz, 2H), 2.45 (t, J = 7.6 Hz, 2H), 2.26 (s, 6H), 1.74 – 1.65 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -54.9 (m, 3F), -81.9 (m, 3F), -81.6 – -84.2 (m, 2F), -83.9 (m, 3F), -126.6 (m, 1F), -147.9 (m, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 162.3 (d, J = 26 Hz), 61.4, 57.8, 45.3, 30.8, carbons corresponding to the $CF_3OCF(CF_3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-EI (m/z) calcd for $C_{12}H_{12}F_{13}NO_4$ $[M+H]^+$: 481.0553, found: 481.0551. IR (film) ν/cm^{-1}: 1695.4, 1667.6, 1411.6, 1232.5, 1159.1, 1042.2, 981.2, 892.9, 821.8, 771.5, 739.3, 683.4, 654.7, 539.2.

N-(2-(dimethylamino)ethyl)-2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(trifluoromethoxy)propoxy)propanamide (**20**) **C72-Amine-E**

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added N,N-dimethylethane-1,2-diamine (20 mmol, 1.76 g, 1 eq.), triethylamine (30 mmol, 3.04 g, 1.5 eq.) and methanol (10 mL). Then, compound $CF_3OCF(CF_3)CF_2OCF(CF_3)COOCH_3$ (**2**, 20 mmol, 8.20 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. After the reaction the solvent was removed by rotary evaporation under vacuum. Dichloromethane (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product obtained as a light-yellow liquid (8.77 g, 94%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.42 (t, J = 6.4 Hz, 2H), 2.47 (t, J = 7.6 Hz, 2H), 2.26 (s, 6H). ^{19}F NMR (376 MHz, CD_3OD) δ -55.0 (m, 3F), -81.0 (m, 3F), -80.4 – -84.9 (m, 2F), -84.3 (m, 3F), -134.0 (m, 1F), -147.8 (m, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 159.5 (d, J = 26 Hz), 58.3, 45.4, 38.8, carbons corresponding to the $CF_3OCF(CF_3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{11}H_{10}F_{13}N_2O_3$ $[M-H]^-$: 465.0489, found: 465.0488. IR (film) ν/cm^{-1}: 3350.1, 2955.1, 2869.2, 2830.1, 2781.8, 1716.0, 1520.0, 1463.6, 1356.2, 1235.5, 1162.2, 1107.5, 1077.4, 1054.8, 981.9, 893.0, 846.7, 808.9, 773.3, 739.1, 684.1, 619.6, 536.0.

N,N-dimethyl-3-(2,3,3,3-tetrafluoro-2-(perfluoropropoxy)propanamido)propan-1-amine oxide (**21**) **C61-Oxide** [10e]

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3CF_2CF_2OCF(CF_3)C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**11**, 5 mmol, 2.07 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 12 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous liquid (2.09 g, 97%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.42 (t, J = 6.8 Hz,2H), 3.32 (m, 2H), 3.16 (s, 6H), 2.15 – 2.07 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -81.8 (dm, J = 149.7 Hz, 1F), -82.9 (t, J = 7.5 Hz, 3F), -84.0 (d, J = 1.9 Hz, 3F), -86.2 (dm, J = 149.3 Hz 1F), -131.1 (m, 2F), -134.0 (dd, J = 19.2, 6.4 Hz, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 159.5 (d, J = 27 Hz), 69.2, 58.6 (d, J = 3 Hz), 38.8, 24.2, carbons corresponding to the $CF_3CF_2CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{11}H_{14}F_{11}N_2O_3$ $[M+H]^+$: 431.0823, found: 431.0813. IR (film) ν/cm^{-1}: 3261.9, 2810.1, 1709.2, 1544.3, 1477.0, 1451.4, 1323.0, 1234.7, 1165.6, 1134.9, 1072.0, 991.6, 927.8, 886.1, 809.2, 747.9, 721.0, 630.3, 536.6.

N,N-dimethyl-3-(2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(trifluoromethoxy)propoxy)propanamido)propan-1-amine oxide (**22**) **C72-Oxide**

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3OCF(CF_3)CF_2OCF(CF_3)C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**12**, 5 mmol, 2.40 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 18 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous liquid (2.43 g, 98%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.43 (t, J = 6.8 Hz,2H), 3.31 (m, 2H), 3.15 (s, 6H), 2.14 – 2.06 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -54.9 (m, 3F), -81.0 (m, 3F), -80.3 – -84.9 (m, 2F), -84.4 (m, 3F), -133.9 (m, 1F), -147.8 (m, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 159.5 (d, J = 26 Hz), 69.2, 58.7 (d, J = 4 Hz), 38.8, 24.2, carbons corresponding to the $CF_3OCF(CF_3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{12}H_{14}F_{13}N_2O_4$ $[M+H]^+$: 497.0741, found: 497.0736. IR (film) ν/cm^{-1}: 3226.1, 2804.3, 1709.6, 1541.4, 1455.9, 1161.7, 1070.9, 1026.3, 981.8, 892.8, 809.2, 773.4, 739.0, 684.0, 644.5, 535.5.

N,N-dimethyl-3-(2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(perfluoroethoxy)propoxy)propanamido)propan-1-amine oxide (**23**) **C82-Oxide**

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3CF_2OCF(CF_3)CF_2OCF(CF_3)C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**13**, 5 mmol, 2.65 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 12 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous liquid (2.68 g, 98%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.39 (t, J = 7.2 Hz, 2H), 3.30 (m, 2H), 3.14 (s, 6H), 2.13 – 2.04 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -81.5 (m, 3F), -80.7 – -84.8 (m, 2F), -84.1 (m, 3F), -87.0 (m, 2F), -88.2 (d, J = 13.9 Hz, 3F), -133.8 (dd, J = 19.9, 7.1 Hz, 1F), -146.4 (t, J = 21.8 Hz, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 159.4 (d, J = 26 Hz), 69.2, 58.6 (d, J = 3 Hz), 38.7, 24.2, carbons corresponding to the $CF_3CF_2OCF(CF_3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{13}H_{14}F_{15}N_2O_4$ $[M+H]^+$: 547.0709, found: 547.0693. IR (film) ν/cm^{-1}: 3250.5, 2806.1, 1708.4,

1541.1, 1476.8, 1455.1, 1307.1, 1234.9, 1153.6, 1090.3, 1070.0, 1026.2, 981.7, 927.9, 885.5, 825.7, 805.9, 765.3, 743.6, 721.4, 691.5, 645.0, 521.9.

N,N-dimethyl-3-(2,3,3,3-tetrafluoro-2-(1,1,2,2-tetrafluoro-2-(trifluoromethoxy)ethoxy)propanamido)propan-1-amine oxide (**24**) **C62-Oxide**

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3OCF_2CF_2OCF(CF_3)C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**14**, 5 mmol, 2.15 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 12 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous liquid (2.14 g, 96%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.41 (t, J = 7.2 Hz, 2H), 3.31 (m, 2H), 3.16 (s, 6H), 2.14 – 2.07 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -57.0 (t, J = 9.6 Hz, 3F), -84.0 (d, J = 2.3 Hz, 3F), -86.3 (dd, J = 146.6, 18.4 Hz, 1F), -90.2 (dd, J = 146.6, 8.3 Hz, 1F), -91.9 (m, 2F), -134.0 (dd, J = 18.4, 7.9 Hz, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 159.6 (d, J = 27 Hz), 69.2, 58.7 (d, J = 3 Hz), 38.8, 24.0, carbons corresponding to the $CF_3OCF_2CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{11}H_{14}F_{11}N_2O_4$ $[M + H]^+$: 447.0772, found: 447.0761. IR (film) ν/cm^{-1}: 3254.2, 2810.0, 1709.0, 1541.3, 1452.0, 1398.6, 1223.4, 1150.1, 1068.4, 1026.8, 984.6, 967.7, 903.0, 795.8, 721.9, 681.3, 616.9.

1,1,1,3,3,5,5,6,6,8-decafluoro-N,N-dimethyl-9-oxo-8-(trifluoromethyl)-2,4,7-trioxa-10-azatridecan-13-amine oxide (**25**) **C73-Oxide**

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3OCF_2OCF_2CF_2OCF(CF_3)C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**15**, 5 mmol, 2.48 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 18 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous liquid (2.48 g, 97%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.44 – 3.34 (m, 4H), 3.21 (s, 6H), 2.14 – 2.04 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -55.2 (m, 2F), -58.7 (t, J = 9.0 Hz, 3F), -84.1 (d, J = 1.9 Hz, 3F), -86.6 (dd, J = 146.3, 18.4 Hz, 1F), -90.2 (dd, J = 146.3, 7.9 Hz, 1F), -91.7 (m, 2F), -134.0 (dd, J = 18.8, 8.7 Hz, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 159.7 (d, J = 27 Hz), 68.9, 58.1 (d, J = 3 Hz), 38.6, 24.1, carbons corresponding to the $CF_3OCF_2OCF_2CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{12}H_{14}F_{13}N_2O_5$ $[M + H]^+$: 513.0690, found: 513.0677. IR (film) ν/cm^{-1}: 3265.4, 2812.7, 1708.2, 1541.2, 1478.5, 1450.9, 1392.3, 1312.0, 1216.0, 1111.3, 1035.5, 964.5, 870.3, 794.5, 721.0, 646.8.

N,N-dimethyl-3-(2,2,3,3,4,4,5,5,6,6,6-undecafluorohexanamido) propan-1-amine oxide (**26**) **C6-Oxide** [13]

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3CF_2CF_2CF_2CF_2C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**16**, 5 mmol, 1.99 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 18 h. The organic extract was concentrated by rotary evaporation under vacuum. The final compound product was obtained as a white viscous liquid (1.97 g, 95%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.51 (t, J = 6.4 Hz, 2H), 3.19 (s, 6H), 2.98 (t, J = 6.4 Hz, 2H), 2.18 – 2.09 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -82.5 (m, 3F), -118.1 (m, 2F), -123.8 (m, 4F), -127.5 (m, 2F). ^{13}C NMR (100 MHz, CD_3OD): δ 162.9 (t, J = 24 Hz), 70.1, 58.8, 39.0, 23.6, carbons corresponding to the $CF_3CF_2CF_2CF_2CF_2$- group cannot be identified due to C-F coupling. IR (film) ν/cm^{-1}: 3058.3, n 1686.2, 1479.1, 1458.8, 1395.4, 1350.5, 1235.6, 1204.6, 1149.5, 1106.9, 1084.1, 1061.8, 994.7, 953.5, 868.5, 827.4, 808.7, 748.0, 732.7, 714.4, 656.0, 566.4, 531.3.

N,N-dimethyl-3-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluorooctanamido)propan-1-amine oxide (**27**) **C8-Oxide** [14]

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3CF_3CF_3CF_2CF_2CF_2CF_2C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**17**, 5 mmol, 2.49 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 18 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous solid (2.42 g, 97%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.48 (t, J = 6.8 Hz, 2H), 3.17 (s, 6H), 2.92 (t, J = 6.8 Hz, 2H), 2.08 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -82.5 (m, 3F), -118.1 (m, 2F), -122.8 (m, 2F), -123.2 (m, 2F), -123.7 (m, 2F), -123.9 (m, 2F), -127.5 (m, 2F). IR (film) ν/cm^{-1}: 3034.9, 1659.9, 1456.4, 1397.9, 1363.0, 1323.1, 1238.3, 1204.2, 1146.2, 1104.5, 1017.9, 815.9, 736.3, 722.7, 666.1, 642.0, 560.5, 530.5.

N,N-dimethyl-2-((2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(trifluoromethoxy)propoxy)propanoyl)oxy)ethan-1-amine oxide (**28**) **C72-O-Oxide-E**

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3OCF(CF_3)CF_2OCF(CF_3)C(O)OCH_2CH_2N(CH_3)_2$ (**18**, 5 mmol, 2.34 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 18 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous solid (2.32 g, 96%). ^{1}H NMR (400 MHz, CD_3OD) δ 4.01 (m, 2H), 3.47 (m, 2H), 3.26 (s, 6H). ^{19}F NMR (376 MHz, CD_3OD) δ -54.9 (m, 3F), -81.5 (m, 3F), -81.6 – -84.1 (m, 2F), -83.7 (m, 3F), -126.5 (m, 1F), -147.8 (m, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 162.4 (m), 72.6, 59.2, 57.5, carbons corresponding to the $CF_3OCF(CF_3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-EI (m/z) calcd for $C_{11}H_{10}F_{13}NO_5$ $[M]^+$: 483.0346, found: 483.0348. IR (film) ν/cm^{-1}: 3427.5, 2820.1, 1689.7, 1401.2, 1235.3, 1158.5, 1092.6, 1040.1, 981.7, 893.2, 820.5, 771.4, 739.4.

N,N-dimethyl-3-((2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(trifluoromethoxy)propoxy)propanoyl)oxy)propan-1-amine oxide (**29**) **C72-O-Oxide**

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3OCF(CF_3)CF_2OCF(CF_3)C(O)OCH_2CH_2CH_2N(CH_3)_2$ (**19**, 5 mmol, 2.41 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 18 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous solid (2.44 g, 98%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.62 (t, J = 6.0 Hz, 2H), 3.41 (t, J = 7.6 Hz, 2H), 3.17 (s, 6H), 2.08 – 1.98 (m, 2H). ^{19}F NMR (376 MHz, CD_3OD) δ -54.9 (m, 3F), -81.5 (m, 3F), -81.6 – -84.2 (m, 2F), -83.8 (m, 3F), -126.6 (m, 1F), -147.9 (m, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 162.3 (m), 70.0, 60.4, 58.5, 28.0, carbons corresponding to the $CF_3OCF(CF_3)CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. IR (film) ν/cm^{-1}: 3237.6, 2807.7, 1694.3, 1405.6, 1232.5, 1157.6, 1092.1, 1039.6, 980.8, 892.5, 820.0, 770.9, 738.8, 682.9, 653.5, 537.3.

N,N-dimethyl-2-(2,3,3,3-tetrafluoro-2-(1,1,2,3,3,3-hexafluoro-2-(trifluoromethoxy)propoxy)propanamido)ethan-1-amine oxide (**30**) **C72-Oxide-E**

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CF_3OCF(CF_3)CF_2OCF(CF_3)C(O)NHCH_2CH_2N(CH_3)_2$ (**20**, 5 mmol, 2.33 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 18 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous liquid (2.41 g, 98%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.79 (t, J = 6.4 Hz, 2H), 3.44 (t, J = 8.0 Hz, 2H), 3.17 (s, 6H). ^{19}F NMR (376 MHz, CD_3OD) δ -54.9 (m, 3F), -81.5 (m, 3F), -84.0 – -84.9 (m, 2F), -84.4 (m, 3F), -134.0 (m, 1F), -147.8 (m, 1F). ^{13}C NMR (100 MHz, CD_3OD): δ 159.5 (m), 68.1, 59.0 (d, J = 4 Hz), 36.1, carbons corresponding to the $CF_3OCF(CF_3)$

$CF_2OCF(CF_3)$- group cannot be identified due to C-F coupling. HRMS-ESI (m/z) calcd for $C_{11}H_{10}F_{13}N_2O_4$ $[M-H]^-$: 481.0438, found: 481.0438. IR (film) ν/cm^{-1}: 3208.5, 2799.1, 1715.5, 1541.4, 1478.1, 1456.5, 1235.3, 1161.9, 1073.4, 1043.9, 982.1, 893.1, 809.1, 773.6, 739.5, 684.1, 620.8, 535.0, 469.9.

N-(3-(dimethylamino)propyl)dodecanamide (**31**) **CH12-Amine** [1a]

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar was added 3-dimethylaminopropylamine (20 mmol, 2.04 g, 1 eq.), chloroform (10 mL). Then, compound $C_{11}H_{23}COCl$ (**9**, 20 mmol, 4.38 g, 1.0 eq.) was slowly dropped in and the mixture was stirred at room temperature for 24 h. After the reaction the solvent was removed by rotary evaporation under vacuum. Dichloromethane (50 mL) was added to the residue. The resulting mixture was washed with saturated brine and water. The organic phase was separated and then dried over anhydrous sodium sulfate. After filtration, the solvent was removed by rotary evaporation under vacuum. The final product was obtained as a white solid (5.12 g, 90%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.18 (t, $J = 6.8$ Hz, 2H), 2.36 (t, $J = 7.6$ Hz, 2H), 2.25 (s, 6H), 2.16 (t, $J = 7.2$ Hz, 2H), 1.75 – 1.53 (m, 4H), 1.36 – 1.21 (m, 18H), 0.89 (t, $J = 6.4$ Hz, 3H). IR (film) ν/cm^{-1}: 3294.1, 2925.0, 2854.2, 2815.3, 2764.3, 1645.5, 1556.4, 1464.8, 1383.9, 1263.3, 1154.9, 1099.6, 1042.0, 721.7.

3-dodecanamido-N,N-dimethylpropan-1-amine oxide (**32**) **CH12-Oxide** [1a]

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CH_3(CH_2)_{10}C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**31**, 5 mmol, 1.42 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 24 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous solid (1.46 g, 97%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.35 – 3.23 (m, 4H), 3.15 (s, 6H), 2.18 (t, $J = 7.6$ Hz, 2H), 2.08 – 1.98 (m, 2H), 1.64 – 1.54 (m, 2H), 1.36 – 1.21 (m, 18H), 0.89 (t, $J = 6.4$ Hz, 3H). IR (film) ν/cm^{-1}: 3331.2, 3103.9, 2915.3, 2848.2, 1643.7, 1545.9, 1472.6, 1462.5, 1425.1, 1403.3, 1375.1, 1360.4, 1328.4, 1306.9, 1273.5, 1250.3, 1232.8, 1224.3, 1210.3, 1121.0, 1066.1, 1027.6, 1009.5, 976.6, 935.5, 864.6, 786.2, 770.5, 754.2, 729.2, 719.6, 689.7, 615.0, 561.6, 514.0, 489.8, 427.8.

N,N-dimethyldodecan-1-amine oxide (**33**) **C12-Oxide** [1b]

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CH_3(CH_2)_{11}N(CH_3)_2$ (**10**, 5 mmol, 1.07 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (15 mmol, 1.70 g, 3.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 24 h. The organic extract was concentrated by rotary evaporation under vacuum. The final product was obtained as a white viscous solid (1.10 g, 96%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.23 (t, $J = 8.4$ Hz, 2H), 3.11 (s, 6H), 1.85 – 1.75 (m, 2H), 1.40 – 1.20 (m, 18H), 0.86 (t, $J = 6.8$ Hz, 3H). IR (film) ν/cm^{-1}: 3372.9, 2921.8, 2852.3, 1652.2, 1467.3, 1378.0, 965.7, 924.8, 773.8, 721.0.

N-(3-(dimethylamino)propyl)stearamide (**35**) **CH18-Amine** [15]

To a 100 mL three-necked round-bottom flask containing a magnetic stirring bar and N_2 were added $C_{17}H_{35}COOH$ (20 mmol, 6.04 g, 1.0 eq.), and toluene (25 mL). Then, compound 3-dimethylaminopropylamine (60 mmol, 6.12 g, 3 eq.) was slowly dropped into the mixture at 75 °C, and the reaction was heated to 130 °C for 24 h. After removing the solvent by rotary evaporation under vacuum. The residue was recrystallized over acetone for twice. The final product was obtained as a white solid (5.24 g, 71%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.18 (t, $J = 6.8$ Hz, 2H), 2.33 (t, $J = 8$ Hz, 2H), 2.26 (s, 6H), 1.72 – 1.54 (t, $J = 7.6$ Hz, 4H), 1.38 – 1.21 (m, 24H), 0.89 (t, $J = 6.4$ Hz, 3H). IR (film) ν/cm^{-1}: 3310.4, 2917.6, 2849.3, 2814.2, 2763.0, 1640.0, 1557.4, 1472.1, 1395.0, 1376.5, 1258.7, 1241.4, 1186.4, 1155.0, 1042.4, 942.3, 729.6, 718.9.

N,N-dimethyl-3-stearamidopropan-1-amine oxide (**36**) **CH18-Oxide** [15].

To a 100 mL round-bottom flask containing a magnetic stirring bar was added $CH_3(CH_2)_{16}C(O)NHCH_2CH_2CH_2N(CH_3)_2$ (**35**, 5 mmol, 1.84 g, 1 eq.), and ethanol (50 mL). The reaction mixture was stirred evenly and heated to 50 °C. Then, hydrogen peroxide (20 mmol, 2.28 g, 4.0 eq.) was slowly dropped in and the mixture was stirred at 50 °C for 12 h. The organic extract was concentrated by rotary evaporation under vacuum. The final compound product was obtained as a white solid (1.87 g, 97%). ^{1}H NMR (400 MHz, CD_3OD) δ 3.31 – 3.21 (m, 4H), 3.15 (s, 6H), 2.16 (t, $J = 8.0$ Hz, 2H), 2.05 – 1.98 (m, 2H), 1.64 – 1.53 (m, 2H), 1.38-1.16 (m, 28H), 0.88 (t, $J = 6.8$ Hz, 3H). IR (film) ν/cm^{-1}: 3325.6, 2916.7, 2849.0, 1643.7, 1550.8, 1467.5, 1378.6, 1274.1, 1258.8, 1242.3, 1223.7, 1208.8, 1123.1, 1066.0, 1010.3, 936.3, 869.4, 720.1, 616.7, 488.3, 427.1.

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi

Declaration of Competing Interest

The authors report no declarations of interest.

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Supplementary materials

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