

(MeFASAs and EtFASAs; Table 3) (3M Company 1999; Lehmler 2005). These *N*-alkyl FASAs are, in some cases, commercial products in their own right, as well as building blocks for further synthesis. For instance, *N*-ethyl perfluorooctane sulfonamide, $C_8F_{17}SO_2NH(C_2H_5)$, or EtFOSA, is the pesticide sulfluramid. In a 2nd major industrial reaction step, *N*-alkyl FASAs are reacted with ethylene carbonate to give another series of building blocks, the *N*-methyl or *N*-ethyl perfluoroalkane sulfonamido ethanols, $C_nF_{2n+1}SO_2N(C_mH_{2m+1})CH_2CH_2OH$, where $m=1$ or 2 (MeFASEs and EtFASEs; Table 3) (3M Company 1999; Lehmler 2005). These *N*-alkyl FASEs are analogous to FTOHs. Because they are alcohols, they can be converted into acrylates and methacrylates, as well as into phosphates and other derivatives (3M Company 1999) that will not be discussed further here. The *N*-alkyl perfluoroalkane sulfonamidoethyl acrylates, $C_nF_{2n+1}SO_2N(C_mH_{2m+1})CH_2CH_2OC(O)CH=CH_2$, where $m=1$ or 2 (MeFASACs and EtFASACs; Table 3) and the corresponding *N*-alkyl perfluoroalkane sulfonamidoethyl methacrylates, $C_nF_{2n+1}SO_2N(C_mH_{2m+1})CH_2CH_2OC(O)C(CH_3)=CH_2$ (MeFASMACs and EtFASMACs; Table 3) are used in a similar manner to the fluorotelomer acrylates and methacrylates, as comonomers for synthesizing acrylic polymers used in surface protection applications (3M Company 1999).

The (alkyl-)FASA, FASE, FASAC, and FASMAC intermediates were the principal building blocks of many fluorochemical products used in surface treatments, paper packaging protectors, and other specialist applications. A more extensive range of commercial compounds has, however, been produced, as described in industry reports (3M Company 1999). In 2002, the largest historic manufacturer of perfluorooctane sulfonyl derivatives ($n \geq 6$) ceased manufacture (3M Company 2000a; USEPA 2000) and has since introduced products based on perfluorobutane sulfonyl

chemistry (Renner 2006; Ritter 2010). Meanwhile, existing and new manufacturers continue to make PFOS and other long-chain perfluoroalkane sulfonates and products derived from them.

Degradation products of perfluoroalkane sulfonamido derivatives: Published studies on the aerobic biotransformation of the perfluoroalkane sulfonamido derivatives focus on those compounds having 8 perfluorinated C atoms, in particular *N*-ethyl perfluorooctane sulfonamidoethanol (EtFOSE), which is ultimately degraded to PFOS. Various intermediates leading to this perfluoroalkane sulfonic acid have been reported, including the members of the following families (Tables 2 and 3) with $n=8$: *N*-ethyl perfluoroalkane sulfonamidoacetic acids (EtFASAAAs), $C_nF_{2n+1}SO_2N(C_2H_5)CH_2COOH$; *N*-ethyl perfluoroalkane sulfonamides (EtFASAs), $C_nF_{2n+1}SO_2NH(C_2H_5)$; perfluoroalkane sulfonamidoacetic acids (FASAAAs), $C_nF_{2n+1}SO_2NHCH_2COOH$; perfluoroalkane sulfonamides (FASAs), $C_nF_{2n+1}SO_2NH_2$; FASA *N*-glucuronides, and perfluoroalkane sulfonic acids (PFSIAs), $C_nF_{2n+1}SO_2H$ (Lange 2000, 2001; Tomy, Tittlemier, et al. 2004; Xu et al. 2004; Boulanger et al. 2005; Xu et al. 2006; Rhoads et al. 2008; Xie et al. 2009) (Figure 8). There appears to be conflicting evidence as to whether PFOA can be formed in the environment from EtFOSE as a minor end product (Lange 2001; Tomy, Tittlemier, et al. 2004; Boulanger et al. 2005; Rhoads et al. 2008).

The *N*-alkyl perfluoroalkane sulfonamidoethyl acrylates and methacrylates, and polymers based on them, may undergo hydrolysis of the ester linkage in the environment to give *N*-alkyl FASEs (Martin et al. 2010) and, hence, lead to the same perfluoroalkyl biotransformation products. However, there do not appear to have been any published experimental studies that explicitly demonstrate this to be the case.

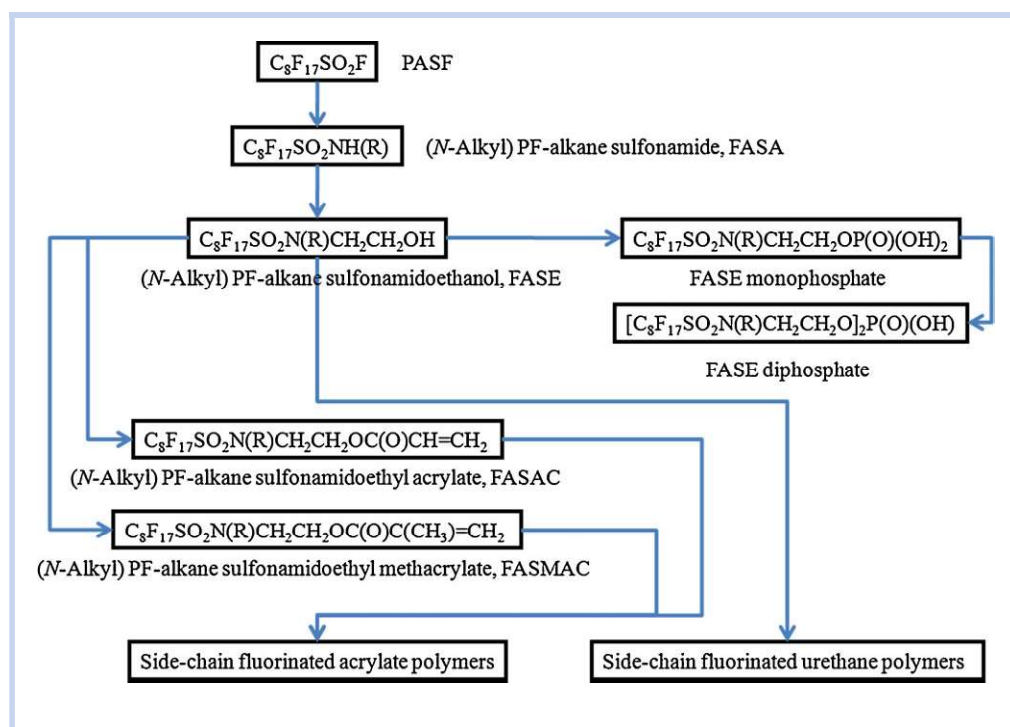


Figure 7. Perfluoroalkane sulfonamido derivatives synthesized from perfluoroalkane sulfonyl fluorides (PASFs), exemplified for a starting PASF with 8 C atoms. N.B. Names and acronyms for substance families are indicated. Those for the specific compounds shown can be found in the Supplemental Data.

Studies on the hydroxyl-radical-initiated degradation of EtFOSE in the aqueous phase show that some of the intermediates and products observed, including EtFOSAA, EtFOSA, FOSAA, FOSA, and PFOA, are the same as those reported for biodegradation. On the other hand, PFOS and PFOSI were not observed or were present at only trace levels in these abiotic studies (Hatfield 2001; Plumlee et al. 2009) and FOSA was considered to be a stable end product (Plumlee et al. 2009).

Atmospheric degradation pathways have been studied for 2 perfluoroalkane sulfonamido derivatives having 4 perfluorinated C atoms. The breakdown of EtFBSA, $C_4F_9SO_2NH(C_2H_5)$, has been shown to proceed through ketone and aldehyde intermediates to give PFCAs, i.e., PFBA, PFPrA and TFA, as well as COF_2 (Martin et al. 2006). The PFPrA and TFA are formed via chain unzipping of the perfluoroalkoxy radical, as already mentioned above for FTOHs and depicted schematically on Figure 6, so that alkyl-FASAs share part of their degradation scheme with FTOHs. PFBS was not observed to be formed from EtFBSA (Martin et al. 2006). MeFBSE, $C_4F_9SO_2N(CH_3)CH_2CH_2OH$, was observed to degrade to the same PFCAs as EtFBSA, together with PFBS, MeFBSA, and other products (D'eon et al. 2006).

Environmental occurrence of perfluoroalkyl sulfonamido derivatives: Various perfluoroalkyl sulfonamido derivatives have been found in the environment and human samples, whether this is due to industrial or consumer use of these compounds as such, losses during manufacturing operations, presence as “residuals” in other commercial products, or formation as environmental degradation products or metabolites of precursors.

It should be noted that perfluoroalkane sulfonamido derivatives bearing a H on the N atom are acidic in nature and can dissociate to an amide anion, to a greater or lesser extent depending on the ambient environmental or physiological conditions, with the degree of branching of the perfluoroalkyl chain having a significant influence on the pK_a for a given family of compounds (Rayne and Forest 2009a). For FASAs, there is the additional possibility of dissociation of the carboxylic H (more acidic than the amide H), whereas for the *N*-alkyl FASAs, this is the only possible ionization (Rayne and Forest 2009a). The dissociated species are not depicted in the list of compounds provided in the Supplemental Data.

All the families of perfluoroalkane sulfonamido derivatives discussed above and depicted in Tables 2 and 3 have been found in the environment or in human biota. Those with 8 perfluorinated C atoms are, in general, much more abundant than those with other chain lengths. However, more recently, compounds with 4 such C atoms have also been reported. The medium in which they are detected depends on their physical properties and on their likelihood of being formed there from precursors. In atmospheric air and its associated particulate matter, commonly detected compounds are the relatively volatile FOSA, MeFBSA, MeFOSA, Me₂FOSA, EtFOSA, MeFBSE, EtFBSE, MeFOSE, and EtFOSE (Martin et al. 2002; Barber et al. 2007; Piekarczyk et al. 2007; Stock et al. 2007; Dreyer, Matthias, et al. 2009; Dreyer, Weinberg, et al. 2009; Dreyer et al. 2010; Langer et al. 2010; Shoeib et al. 2010; Haug et al. 2011; Weinberg et al. 2011a, 2011b), whereas house dust has been found to contain FOSA, MeFOSA, EtFOSA, MeFOSE, EtFOSE (Shoeib et al. 2005; Kato et al. 2009; Goosey and Harrad 2011), the acrylate MeFOSAC (Shoeib et al. 2005), and the oxidation products MeFOSAA and EtFOSAA (Kato et al. 2009). FOSA has also

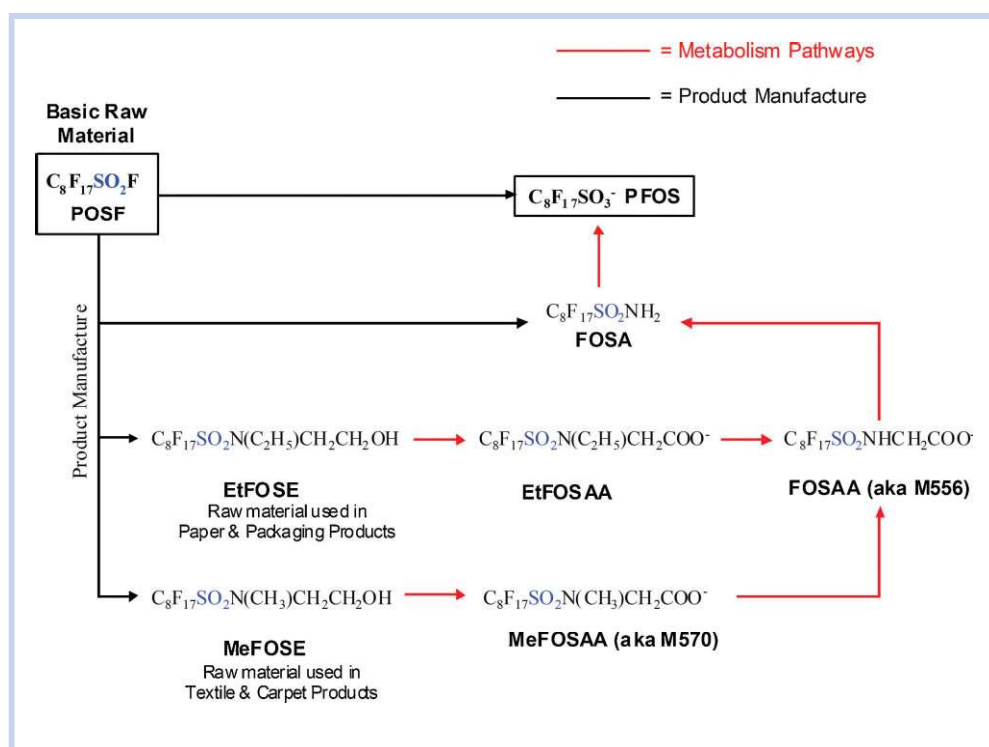


Figure 8. Transformation pathways for perfluoroalkane sulfonamido derivatives. Adapted from Olsen et al. 2002 and Olsen et al. 2005.

been detected in open ocean water, sometimes at levels comparable to those of PFOA (Ahrens, Gerwinski, et al. 2010; Ahrens, Xie, et al. 2010; Busch et al. 2010b; Kirchgeorg et al. 2010), as well as in precipitation (Kim and Kannan 2007; Taniyasu et al. 2008; Kwok et al. 2010), river and lake water (Kim and Kannan 2007; So et al. 2007; Ahrens et al. 2009b; Ahrens, Gerwinski, et al. 2010; Scott et al. 2010; Zushi et al. 2011), groundwater (Murakami, Kuroda, et al. 2009), surface runoff water (Kim and Kannan 2007; Murakami, Shinohara, et al. 2009), landfill leachate (Kallenborn et al. 2004; Busch et al. 2010a; Huset et al. 2011), sewage sludge (Llorca et al. 2011), and drinking water (Ericson et al. 2009). In wildlife, FOSA is often the predominant sulfonamido species, although it is generally present at lower levels than PFOS (Sturm and Ahrens 2010 and references therein), whereas EtFOSA and/or Et₂FOSA (Tomy, Budakowski, et al. 2004; Tittlemier et al. 2005; Tittlemier et al. 2006; Löfstrand et al. 2008; Ahrens, Siebert, et al. 2009; Yeung et al. 2009), MeFOSE (Ahrens and Ebinghaus 2010), FOSAA (Peng et al. 2010), and EtFOSAA (Yoo et al. 2009) have also been reported. FOSA and various *N*-alkyl-FOSAs (Me-, Et-, Me₂-, and Et₂-FOSAs) were detected in foodstuffs (Tittlemier et al. 2005; Tittlemier et al. 2006). WWTP effluents and river, coastal, and ocean waters were found to contain some *N*-alkyl sulfonamido derivatives (MeFBSA, MeFBSE, MeFOSE, EtFOSE, MeFB-SAA, MeFOSAA, and EtFOSAA) as well as FOSA and FOSAA (Ahrens et al. 2009a; Ahrens et al. 2009b; Ahrens, Gerwinski, et al. 2010; Huset et al. 2011; Nguyen et al. 2011; Zushi et al. 2011). In human blood, the sulfonamido derivatives FOSA, FOSAA, MeFOSAA, and EtFOSAA have been quantified (Kannan et al. 2004; Calafat et al. 2007; Olsen et al. 2008; Weihe et al. 2008; Toms et al. 2009; Lee and Mabury 2011). MeFOSAA and/or EtFOSAA have also been detected in precipitation (Taniyasu et al. 2008; Kwok et al. 2010), wildlife (Yoo et al. 2009), sediments (Higgins et al. 2005; Ahrens, Taniyasu, et al. 2010; Zushi et al. 2010) and WWTP influent and effluent (Boulanger et al. 2005). These 2 compounds have also been shown to be among the most abundant PFAS components of municipal WWTP sludge (Higgins et al. 2005; Sepulvado et al. 2011), in which FOSAA has also been detected (Higgins et al. 2005).

Perfluoroalkyl and polyfluoroalkyl ether carboxylic acids. Salts of perfluoroalkyl ether carboxylic acids (not depicted in the tables) and polyfluoroalkyl ether carboxylic acids (Table 3) are widely cited in patents as alternative fluoropolymer processing aids, that are more environmentally and/or toxicologically acceptable alternatives to APFO and APFN. A common feature is that a terminal -COO⁻ group, attached to one or both ends of the fluorinated ether chain, is the common hydrophile, generally with an NH₄⁺ counter-ion (Tsuda et al. 2003; Visca et al. 2003; Higuchi et al. 2005; Hintzer et al. 2005; Brothers et al. 2008; Ishikawa et al. 2008; Gordon 2011). These and/or other alternative surfactants are expected to enable manufacturers to meet the USEPA 2010/15 Stewardship Program goal to eliminate the use of PFOA and higher homologues. Most recently, a toxicological evaluation for one of these substances (ammonium 4,8-dioxa-3*H*-perfluorononanoate; Table 3) has been published (Gordon 2011). Substances based on certain members of this family of compounds have a sufficient number of repeating units (together with other characteristics) to enable them to

be considered to be polymers under the European Union REACH legislation (ECHA 2008).

Fluorinated polymers

The polymers discussed in this section are those: 1) whose synthesis involves the incorporation of one or more PFASs as monomers. In this case, there is some potential (theoretical or demonstrated) for the degradation of the polymer, during or after its useful lifetime, to lead to release of PFASs to the environment; or 2) whose manufacture requires the use of a PFAS as a processing aid.

Fluoropolymers. Fluoropolymers contain F bound to one or both of the olefinic C atoms, to form a perfluorinated C-only polymer backbone with F atoms directly attached to it (Table 4). Examples of fluoropolymers are polytetrafluoroethylene (PTFE); polyvinylidene fluoride (PVDF); polyvinyl fluoride (PVF); copolymers of tetrafluoroethylene (TFE) and hexafluoropropylene (HFP); terpolymers of TFE, vinylidene fluoride, and HFP; and copolymers of TFE and ethylene. Certain grades of fluoropolymers, manufactured by emulsion (or dispersion) polymerization, in order to obtain a fine particle size distribution, require the use of a fluorosurfactant “processing aid.” This additive, used at a level of a few tenths of a percent relative to the amount of polymer produced (Prevedouros et al. 2006), was often traditionally the ammonium salt of PFOA or PFNA. The fluorosurfactant is removed when the fluoropolymer aqueous emulsion is dried for sale as a solid. Similarly, when an aqueous fluoropolymer emulsion is used, the polymer is heated to cure it. High cure temperatures thermally destroy the fluorosurfactant. At low cure temperatures, residual surfactant may remain (Guo et al. 2009). Most producers have discontinued the use of PFOA and PFNA salts as processing aids and have developed and implemented more environmentally acceptable alternatives, as discussed above in the *Perfluoroalkyl and Polyfluoroalkyl Ether Carboxylic Acids* section. It should be emphasized that those grades of fluoropolymers (e.g., PTFE, PVDF) that are made by suspension (rather than emulsion) polymerization do not require a fluorosurfactant to be used as a “processing aid.”

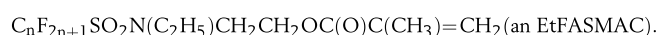
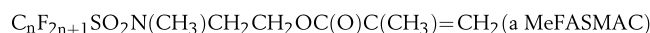
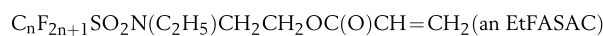
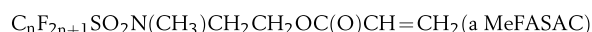
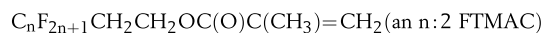
Perfluoropolyethers. Perfluoropolyethers (PFPEs; Table 4) are polymers in whose backbone -CF₂-, -CF₂CF₂-, and possibly -CF(CF₃)CF₂- units are separated by O atoms. For example, the ultraviolet-initiated copolymerization of TFE with O₂ leads to PFPEs with a structure that may be represented symbolically by CF₃O(CF₂CF₂O)_m(CF₂O)_nCF₃, although this overall formula does not show that the -CF₂O- and -CF₂CF₂O- units are generally distributed randomly rather than in blocks (Sianesi et al. 1994). If the photopolymerization is conducted using hexafluoropropylene (HFP) instead of (or together with) TFE, then PFPEs with the overall formula CF₃O(CF₂CF₂O)_m(CF₂O)_n[CF(CF₃)CF₂O]_pCF₃ are obtained. Furthermore, the PFPE -[CF(CF₃)CF₂O]_n- can be synthesized by homopolymerization of HFP (ep)oxide.

Because the repeating units of these PFPEs contain only 2 or 3 perfluorinated C atoms per O atom, their degradation cannot lead to the formation of long-chain PFCAs. The reason for mentioning them in this review is that certain difunctional polymeric perfluoro-polyether products, corresponding to the

overall formula $X-CF_2O(CF_2CF_2O)_m(CF_2O)_nCF_2-X$, where X is a hydrophilic group, are marketed as surface treatments for natural stone, metal, glass, plastic, textiles, leather, and paper and paperboard treatment for food-contact applications. These functionalized PFPEs bring properties such as a low surface energy, high contact angle, reduced coefficient of friction, and high oleo-hydrophobicity (Solvay Solexis 2011), so that they are potential alternatives to the ECF-based polymers, fluorotelomer-based polymers, and fluorinated oxetane polymers described in this review.

Side-chain-fluorinated polymers. In contrast to the polymers described previously, side-chain-fluorinated polymers do not have perfluorinated or polyfluorinated polymer backbones, but are composed of variable composition backbones with polyfluoroalkyl (and possibly perfluoroalkyl) side chains (Table 4). With regard to the sources of long-chain PFAAs, we review 3 groups of side-chain-fluorinated polymers distinguished from one another by the linkage (acrylate and/or methacrylate, urethane, and oxetane) between the polymer backbone and the polyfluoroalkyl (and possibly perfluoroalkyl) side chains. Side chains of each of these polymer types may possess the ability to sever from the polymer chain to become PFASs shown in Tables 2 and 3. It should be noted, however, that this transformation process can occur over long time periods (e.g., >1000 y) and may exhibit low yields of PFASs such that their contribution to the environmental inventory of long-chain PFAAs may be insignificant relative to other historical and current sources. Further research is required to clarify this question.

Fluorinated acrylate polymers: Fluorinated acrylate polymers are made by polymerizing a fluorinated acrylate (or methacrylate) monomer, in which the alcohol moiety is $n:2$ FTOH, $C_nF_{2n+1}CH_2CH_2OH$, or an alkyl-FASE, $C_nF_{2n+1}SO_2N(R)CH_2CH_2OH$, where $R = CH_3$, C_2H_5 , or another alkyl group (Table 4). Some possible structures for the fluorinated acrylate monomers are therefore:



These fluorinated acrylate monomers are copolymerized with one or more nonfluorinated acrylate monomers, and possibly other monomers, to give the final side-chain fluorinated acrylate polymers. These types of polymers are useful as water-, stain- and grease-proofing finishes for textile, leather, and paper surfaces. As stated above, it is not yet clear to what extent such polymers may break down in the environment to give PFAAs, such as PFOA, PFOS, PFBA, and PFBS. Moreover, although we have shown only fluorotelomer and perfluoroalkane sulfonamido (meth)acrylates, the term “side-chain-fluorinated polymer” would encompass many other potential structures and products therefrom that conform to the definition provided.

Fluorinated urethane polymers: Polymeric materials for repelling water and stains may also be based on urethane polymers formed by reacting fluorotelomer alcohols (FTOHs), or perfluoroalkane sulfonamidoethanols (alkyl-FASEs), with polyisocyanato homopolymers, followed by a cross-linking step (Kirchner 1989). The products are polyfluorinated in their side chains (Table 4). They are used mainly in textile applications. In the case of an (8:2) FTOH-based urethane polymer, a recent study has shown that the half-life with respect to biodegradation to PFOA in aerobic soils is on the order of a century (Russell et al. 2010).

Fluorinated oxetane polymers: An alternative fluorinated polymer technology to those described thus far originates from the reaction of polyfluorinated alcohols with oxetanes bearing a $-CH_2Br$ group in their side chains, to create oxetane monomers that can undergo ring-opening polymerization to give side-chain-polyfluorinated polyethers (Figure 9). These fluorinated oxetane polymers (Table 4) are offered in many forms and functionalities primarily as fluorosurfactants and coatings additives (Kausch et al. 2002; Kausch et al. 2003a, 2003b; Thomas 2006; Omnova Solutions 2011).

Commercial articles containing multiple types of fluorinated polymers. It should be noted that there are commercial products that contain both fluoropolymers and side-chain-fluorinated polymers, which can cause confusion about the origin of individual PFASs. In all-weather clothing products, for example, multiple layered materials containing different types of polymers are common. A porous PTFE membrane layer is often used in garments to make the fabric “breathable.” The outer fabric layer may be nylon or polyester

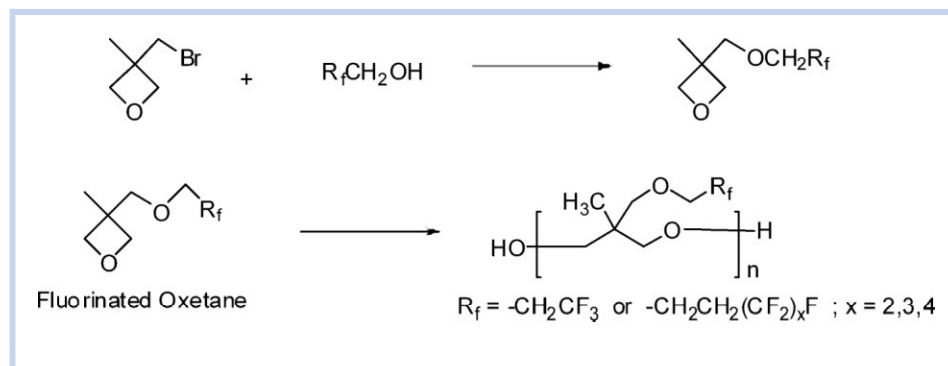


Figure 9. Oxetane-based fluorinated polymers.

treated with a side-chain-fluorinated polymer water repellent. Analyses of all-weather clothing revealed the presence of FTOHs in the outer layer of some all-weather clothing products (Berger and Herzke 2006; Schulze and Norin 2006). The origin of the FTOHs is not the PTFE breathable membrane.

SUMMARY AND FUTURE PROSPECTS

We have provided an overview of PFASs detected in the environment, wildlife, and humans and recommended clear, specific, and descriptive terminology, names, and acronyms for PFASs. We hope the terminology will be widely adopted and used. Future interest in fluorinated substances by the global scientific community is expected to remain high, and continued publications should be numerous. The consistent use of the terminology described here by this community will facilitate clear and coherent communication, understanding, interpretation, and comparison of published studies as well as serve to highlight similarities and acknowledge key differences between PFASs. We strongly discourage the use of broad, poorly defined terms and acronyms in favor of the clear, specific, and descriptive terminology provided here.

SUPPLEMENTAL DATA

Supplemental Data. Names, formulas, acronyms, and CAS numbers for selected perfluoroalkyl and polyfluoroalkyl substances. Terminology decision flow charts.

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(NOTE: For patents, the years indicated are those of filing of the earliest priority application.)

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