



### Objectives:

- To develop methods to determine the concentrations of Per- and Polyfluoroalkyl substances (PFAS) in a variety of food items utilizing samples from FDA Total Diet Study program .
- To assess the impact of food sources in close proximity to environmental inputs of PFAS chemicals – Investigate location in close proximity to PFAS production facility and location close to Air Force Base where groundwater has been contaminated with Aqueous Film Fighting Foam (AFFF).

### Method

Weigh out homogenized food sample

- Add water and extraction solvent (Acetonitrile)
- Add QuEChERS salt (6000 mg MgSO<sub>4</sub> and 1500 mg NaCl)
- Transfer supernatant to dSPE tube (400 mg PSA, 400 mg C18 and 1200 mg MgSO<sub>4</sub>)

#### Shake/Centrifuge Step



Vortex/Shake 5 minutes at 1500 RPM

Centrifuge for 5 minutes at 10,000 x g



4. Filter extract with a 0.2 µm nylon filter



5. Analysis using SCIEX 6500 plus

#### Target Analytes

PFBA	Perfluoro-n-butanoic acid
PFPeA	Perfluoro-n-pentanoic acid
PFHxA	Perfluoro-n-hexanoic acid
PFHpA	Perfluoro-n-heptanoic acid
PFOA	Perfluoro-n-octanoic acid
PFNA	Perfluoro-n-nonoic acid
PFDA	Perfluoro-n-decanoic acid
PFBS	Potassium perfluoro-1-butanesulfonate
PFPeS	Sodium perfluoro-1-pentanesulfonate
PFHxS	Sodium perfluoro-1-hexanesulfonate
PFHpS	Sodium perfluoro-1-heptanesulfonate
PFOS	Sodium perfluoro-1-octanesulfonate
NaDONA	Sodium dodecafluoro-3H-4,8-dioxanonanoate
HFPO-DA	Tetrafluoro-2-(heptafluoropropoxy)propanoic acid
9Cl-PF3ONS	Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate
11Cl-PF3OUdS	Potassium 11-chloroheptafluoro-3-oxaundecane-1-sulfonate

### Total Diet Study Samples (TDS)

TDS samples were used because of the diverse food categories (produce, meat, dairy, and grain products) and they were already prepared for analysis.

- Samples were collected in October 2017 from the Mid-Atlantic Region (see map).
- Analytical sample is a composite of 3 cities per collection in that region of each food item.
- 91 samples were analyzed, including produce, meat, dairy, and grain products.
- Non-detects (16 analytes) were reported in 81 of the 91 samples analyzed.
- Using our current safety assessment methods, the samples with PFOS are not likely to be a health concern.



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ppt (ng/kg) LLOQ ranges from 2 ng/kg to 1000 ng/kg depending on compound and commodity

	PFOA	PFOS	PFBA	PFHpS	PFPeA	PFHxA	PFHxS	PFHpA	PFBS	PFPeS	NaDONA	HFPO-DA	PFDA	PFNA	11Cl-PF3OUdS	9Cl-PF3ONS
Fruits/Vegetables 2 detects in 39 samples																
Pineapple	< LLOQ	< LLOQ	68.4	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Sweet Potato	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	5.2	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Breads/baked goods 1 detect in 16 samples																
Chocolate cake with icing	< LLOQ	< LLOQ	< LLOQ	< LLOQ	17640	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Dairy 1 detect in 12 samples																
Chocolate milk	< LLOQ	< LLOQ	< LLOQ	< LLOQ	154	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Meat/Seafood 10 detects in 21 samples																
Ground Turkey	< LLOQ	765	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Tilapia	< LLOQ	865	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	145	< LLOQ	158
Cod	< LLOQ	192	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Beef steak	< LLOQ	149	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Frankfurter	< LLOQ	134	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Salmon	< LLOQ	253	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Lamb chop	< LLOQ	216	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Shrimp	< LLOQ	676	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Chicken thigh	< LLOQ	180	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ
Catfish	< LLOQ	673	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	< LLOQ	105
Miscellaneous 0 detects of 3 samples																

Dairy Farm near Air Force Base in New Mexico

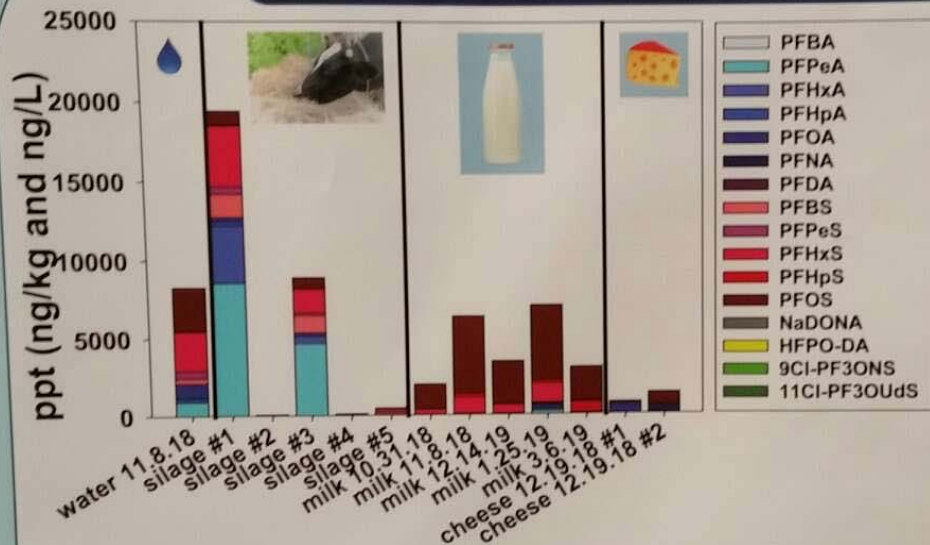
Produce collected near a PFAS Production Plant in Eastern US



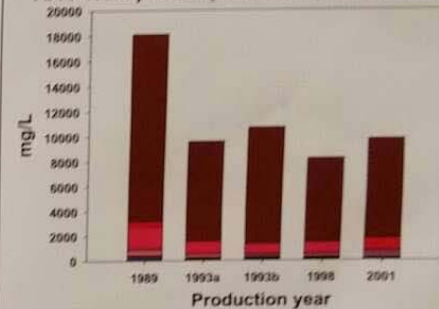
Grown within 10 miles of PFAS Production Plant



# Dairy Farm near Air Force Base in New Mexico



AFFF foam profiles (data from Backe et al. 2013)



- A dairy farm in New Mexico was found to have contaminated groundwater due to its close proximity to an Air Force Base where AFFFs have been historically and currently used.
- The water sample had a PFOS concentration around 2500 ng/L, which is 35 times greater than the EPA health advisory level of 70 ng/L.
- The impact of the water contamination also resulted in PFAS contaminated silage produced in the area.
- As a result, dairy cows were exposed to contaminated water and silage, resulting in milk contamination.
- PFOS has a slow elimination rate from milk even after exposure is stopped. With a half-life of 56 days, it would take 1.5 years to eliminate PFOS from the cow after a 30 day exposure period (Asselt et al. Food Chem. 2013).
- The profiles of PFAS in milk are similar to profiles of AFFF foams reflecting the long term exposure of these chemicals to the cows and the persistence of the long chain compounds in these animals.



- Produce (mainly le... from above and be...)
- Based on previous... contaminated water... (2014).
- Wells near the PFA... from a produce sta...

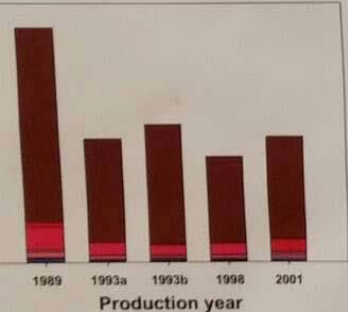
## Conclusions:

PFAS... results for PFOS/PFOA in top commodities using a safety assessment based on food consumption

in

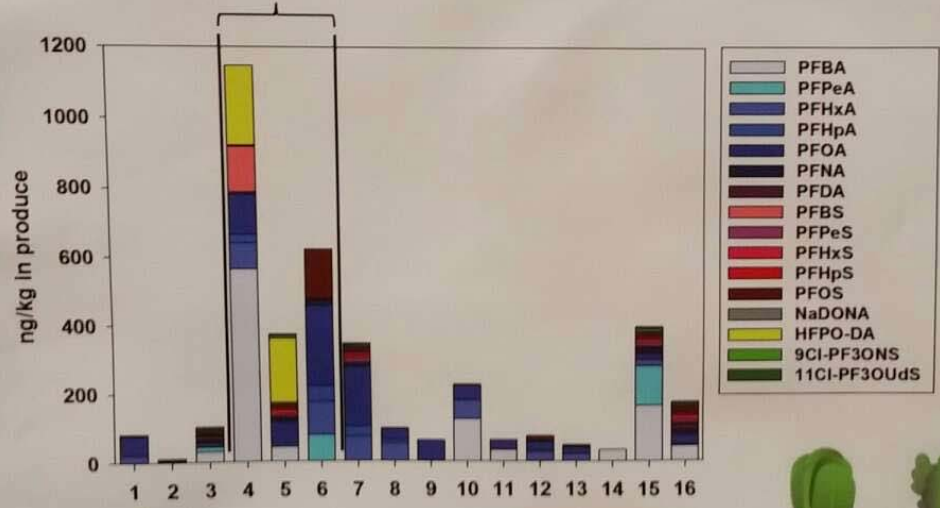
# Produce collected near a PFAS Production Plant in Eastern US

Profiles (data from Backe et al. 2013)



Close proximity to an Air Force Base  
 higher than the EPA health advisory  
 produced in the area.  
 milk contamination.  
 half-life of 56 days, it would take 1.5  
 (J. Environ. Health Chem. 2013).  
 from exposure of these chemicals to

Grown within 10 miles of PFAS Production Plant



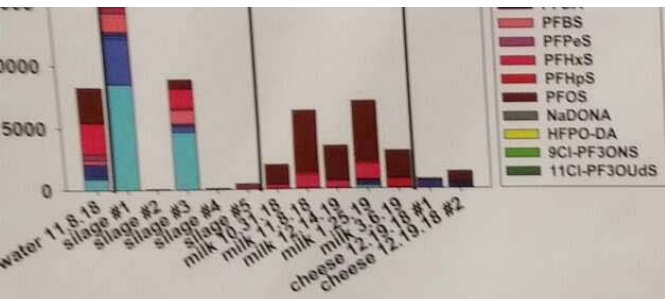
C&E News  
<https://cen.acs.org/articles/96/i7/whats-genx-still-doing-in-the-water-downstream-of-a-chemours-plant.html>



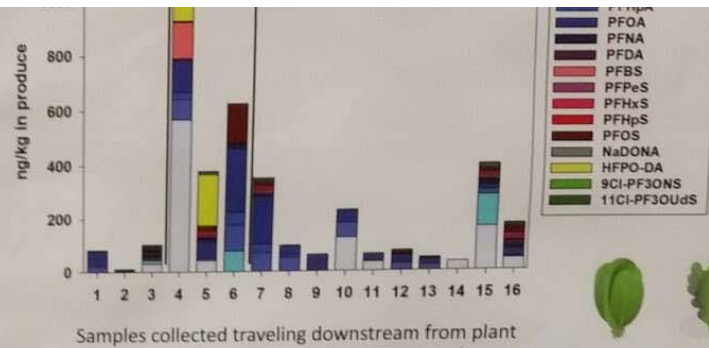
Samples collected traveling downstream from plant

- Produce (mainly lettuce, cabbage, kale, collard greens) were collected at local farmer's markets along the river from above and below the PFAS production plant in June 2018.
- Based on previous studies, longer chain PFAS compounds can uptake into the leafy portion of the plant from contaminated water use, while only the shorter chain compounds will uptake into the fruit. (Blaine et al. ES&T 2014).
- Wells near the PFAS production plant are known to be contaminated with Gen-X (HFPO-DA) and samples collected from a produce stand within 10 miles of the plant had HFPO-DA concentrations ~ 200 ng/kg.





- A dairy farm in New Mexico was found to have contaminated groundwater due to its close proximity to an Air Force Base where AFFFs have been historically and currently used.
- The water sample had a PFOS concentration around 2500 ng/L, which is 35 times greater than the EPA health advisory level of 70 ng/L.
- The impact of the water contamination also resulted in PFAS contaminated silage produced in the area.
- As a result, dairy cows were exposed to contaminated water and silage, resulting in milk contamination.
- PFOS has a slow elimination rate from milk even after exposure is stopped. With a half-life of 56 days, it would take 1.5 years to eliminate PFOS from the cow after a 30 day exposure period (Asselt et al. Food Chem. 2013).
- The profiles of PFAS in milk are similar to profiles of AFFF foams reflecting the long term exposure of these chemicals to the cows and the persistence of the long chain compounds in these animals.



C&E News  
<https://cen.acs.org/articles/PFAS/17/4/11>  
[city-gens-poll-doms-in-the-water](https://cen.acs.org/articles/PFAS/17/4/11)  
[downstream-of-a-chemicals-](https://cen.acs.org/articles/PFAS/17/4/11)  
[plant.html](https://cen.acs.org/articles/PFAS/17/4/11)



- Produce (mainly lettuce, cabbage, kale, collard greens) were collected at local farmer's markets along the river from above and below the PFAS production plant in June 2018.
- Based on previous studies, longer chain PFAS compounds can uptake into the leafy portion of the plant from contaminated water use, while only the shorter chain compounds will uptake into the fruit. (Blaine et al. ES&T 2014).
- Wells near the PFAS production plant are known to be contaminated with Gen-X (HFPO-DA) and samples collected from a produce stand within 10 miles of the plant had HFPO-DA concentrations ~ 200 ng/kg.

**Conclusions:**

- The FDA has reviewed results for PFOS/PFOA in top commodities using a safety assessment based on food consumption data and the EPA reference doses. Use of the developed method and a robust sampling plan will provide a better understanding of potential dietary exposure to consumers that might include TDS and other sampling assignments.
  - Safety assessment was used to advise the New Mexico dairy farm that their milk was unfit for human consumption, the product was discarded.
- PFAS concentrations measured in lettuce and other produce grown near a PFAS production plant were not likely a human health concern from consumption.
  - Results indicate PFAS concentrations measured in produce samples and TDS samples were not likely a human health concern from consumption.
- Water sources and foods grown near potentially contaminated sites will be monitored to ensure the safety of foods being introduced into commerce in the US.

# Investigation of Per- and Polyfluoroalkyl Substances (PFAS) in US food products



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## Background

**PFDA**

- Difficult phased out PFDA completely in 2015.
- Gain in PFPO-DG, a new technology used to make high performance fluoropolymers without PFDA.
- Production has shifted towards shorter chain length perfluorinated substances (PFAEs or PFAA) and other classes of perfluorinated substances.

PFASs are found in:

- Household exposures: Teflon-coated cookware, food packaging, etc.
- Environmental exposures: Air, water, soil.

Dietary intake exposure from agriculture, seafood, and food/water

**PFOS**

- 3M phased out the use of PFOS in 2002.
- PFOS was the key ingredient in producing aqueous film fighting foam (AFFF).
- PFOS was the key ingredient in producing Scotchguard.
- PFOS was replaced with PFAS (6-carbon chain) in producing Scotchguard products.
- Production has shifted towards shorter chain length PFAS.

**PFBS**

**Objectives:**

- To develop methods to determine the concentrations of Per- and Polyfluoroalkyl substances (PFAS) in a variety of food items utilizing samples from FDA Total Diet Study program.
- To assess the impact of food sources in close proximity to environmental inputs of PFAS chemicals – investigate location in close proximity to PFAS production facility and location close to Air Force Base where groundwater has been contaminated with Aqueous Film Fighting Foam (AFFF).

## Method

Method steps:

1. Weigh out, homogenized food sample
2. Add surfactant (e.g., SDS) and stir
3. Extract with hexane
4. Filter extract with 0.2 µm nylon filter
5. Analyze using LC/MS MS/MS plus

Target Analytes:

PFAS	Retention Time (min)
Perfluoro-n-octanoic acid	4.50
Perfluoro-n-decanoic acid	5.50
Perfluoro-n-dodecanoic acid	6.50
Perfluoro-n-tetradecanoic acid	7.50
Perfluoro-n-hexadecanoic acid	8.50
Perfluoro-n-octadecanoic acid	9.50
Perfluoro-n-eicosanoic acid	10.50
Perfluoro-n-docosanoic acid	11.50
Perfluoro-n-tetracosanoic acid	12.50
Perfluoro-n-hexacosanoic acid	13.50
Perfluoro-n-octacosanoic acid	14.50
Perfluoro-n-triacontanoic acid	15.50
Perfluoro-n-dotriacontanoic acid	16.50
Perfluoro-n-tetracontanoic acid	17.50
Perfluoro-n-pentacosanoic acid	18.50
Perfluoro-n-hexacosanoic acid	19.50
Perfluoro-n-heptacosanoic acid	20.50
Perfluoro-n-octacosanoic acid	21.50
Perfluoro-n-nonacosanoic acid	22.50
Perfluoro-n-dotriacontanoic acid	23.50
Perfluoro-n-tetracontanoic acid	24.50
Perfluoro-n-pentacontanoic acid	25.50
Perfluoro-n-hexacontanoic acid	26.50
Perfluoro-n-heptacontanoic acid	27.50
Perfluoro-n-octacontanoic acid	28.50
Perfluoro-n-nonacosanoic acid	29.50
Perfluoro-n-dotriacontanoic acid	30.50
Perfluoro-n-tetracontanoic acid	31.50
Perfluoro-n-pentacontanoic acid	32.50
Perfluoro-n-hexacontanoic acid	33.50
Perfluoro-n-heptacontanoic acid	34.50
Perfluoro-n-octacontanoic acid	35.50

## Total Diet Study Samples (TDS)

TDS samples were used because of the diverse food categories produced, meat, dairy, and grain products) and they were already prepared for analysis. Samples were collected in October 2017 from the Mid-Atlantic Region (see map). Analytical samples in a composite of 3 days per collection in that region of each food item. 50 samples were analyzed, including produce, meat, dairy, and grain products. Non-detects (ND) analyzed were reported in 82 of the 50 samples analyzed. Using our current safety assessment methods, the samples with PFOS are not likely to be a health concern.

PFAS	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-octanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-decanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-dodecanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-tetradecanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-hexadecanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-octadecanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-eicosanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-docosanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-tetracosanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-hexacosanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-octacosanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-dotriacontanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-tetracontanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-pentacontanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-hexacontanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-heptacontanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-octacontanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-nonacontanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoro-n-decacontanoic acid	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

## Dairy Farm near Air Force Base in New Mexico

A dairy farm in New Mexico was found to have contaminated groundwater due to it's close proximity to an Air Force Base where AFFF has been historically and currently used.

The water sample had a PFOS concentration around 2300 ng/L, which is 35 times greater than the EPA health advisory level of 66 ng/L.

The impact of the water contamination did result in PFOS contaminated sheep produced in the area.

As a result, dairy cows were exposed to contaminated water and sludge, resulting in milk contamination.

PFOS has a slow absorption rate from milk even after exposure is stopped. With half life days it would take 2.2 years to eliminate PFOS from the cow after a 30 day exposure period (Smith et al., Food Chem., 2013).

The profiles of PFAS in milk are similar to profiles in sheep following the long term exposure of these chemicals to the feed and the persistence of the long chain compounds in those animals.

## Produce collected near a PFAS Production Plant in Eastern US

Produce (leafy lettuce, cabbages, kale, collard greens) were collected at local farmer's markets along the river from above and below the PFAS production plant in June 2016.

Based on previous studies, longer chain PFAS compounds can uptake into the leafy portion of the plant from contaminated water use, while only the shorter chain counterparts will uptake into the fruit. (Blaine et al. ES&T 2014).

Wells near the PFAS production plant are known to be contaminated with Gen X (PFPO-DG) and samples collected from a produce stand within 10 miles of the plant had PFPO-DG concentrations ~ 200 ng/Lg.

**Conclusions:**

- The FDA has reviewed results for PFOS/PFOA in top commodities using a safety assessment based on food consumption data and the EPA reference doses. Use of the developed method and a robust sampling plan will provide a better understanding of potential dietary exposure to consumers that might include TDS and other sampling assignments.
- Safety assessment was used to address the New Mexico dairy farm that their milk was unfit for human consumption, and the PFAS concentrations measured in lettuce and other produce grown near a PFAS production plant were not likely a human health concern from consumption.
- Results indicate PFAS concentrations measured in produce samples and TDS samples were not likely a human health concern from consumption.
- Water sources and foods grown near potentially contaminated sites will be monitored to ensure the safety of foods before introduced into commerce in the US.