Widespread Occurrence of Non-Extractable Fluorine in Artificial Turfs from Stockholm, Sweden

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**Extraction of samples**

Approximately 1g of each sample was added to a 13 mL PP tube together with 7 mL of acetonitrile. Thereafter, the sample was vortexed and ultrasonicated for 15 minutes. After centrifugation (3500 rpm, 10 minutes), the supernatant was transferred to a new tube, and 6 mL of acetonitrile were added to the samples and the process was repeated. The extracts were combined and concentrated to ~1 mL under nitrogen, then transferred to 1.7 mL Eppendorf centrifuge tubes containing 25 mg of ENVI-Carb™ and 50 μL of glacial acetic acid for clean-up. Extracts were vortexed, centrifuged (10’000 rpm, 10 minutes) and split into clean Eppendorf tubes for CIC (500 μL) and LC-MS analysis (250 μL). Finally, 250 μL of 4 mM NH₄OAc in water and internal standards (ISTD, 1 ng) were added to the extracts for LC-MS analysis and all extracts were stored in the fridge until the day of analysis.

**CIC Analysis**

Prior to analysis, the sample carriers (ceramic boats) were cleaned by burning them at approximately 1100 °C. Each run started and finished with a calibration curve (100 μL of 50 μg/L to 25 mg/L of F⁻ solution, resulting in 5 to 2500 ng total injected) and a mid-level calibration standard was run repeatedly to monitor instrumental drift. Samples for TF analysis ranged from 0.01 to 0.1 g, while for EOF analysis 100 μL of extract were used. The mean fluoride concentration in the method blanks was subtracted from the samples in all cases. The limit of detection (LOD) for total fluorine (TF) analysis was defined as three times the standard deviation in the method blanks (388 ng/g). The LOD for extractable organic fluorine (EOF) analysis was defined in the same way and it was (22.4 ng/g).

**LC-MS/MS Analysis**

PFAS analysis was done on an Aquity ultra-performance liquid chromatograph coupled to a Xevo TQ-S tandem mass spectrometer (UPLC-MS/MS, Waters). Chromatographic separation was achieved injecting 5 μL onto an Acquity UPLC® BEH C18 Column (2.1 × 50 mm, 1.7 μm particle size) plus an Acquity UPLC BEH C18 VanGuard pre-column (2.1 × 5 mm, 1.7 μm particle size) from Waters. A mobile phase gradient using (A) 95% Milli-Q water and 5% acetonitrile with 2 mM ammonium acetate and (B) 95% acetonitrile and 5% Milli-Q water with 2 mM ammonium acetate started with 10% B,
increased to 100% B at 5 min, held constant for 3 min and equilibrated at 10% B for another 2 min. The electrospray ionisation source was operated in negative mode where the source and desolvation temperature were held at 150 °C and 350 °C, respectively, while the capillary voltage was set to 1 kV. Instrumental parameters were based on the method used in a previous study. The monitored ion transitions for all PFAS can be found in Table S2. Quantification was performed using isotope dilution with an 8-point calibration curve (linear, 1/x weighting). Exactly matched, isotopically-labelled standards were used when available, otherwise a structurally similar isotopically-labelled standard was used (see Table S 3). The Limit of Detection (LOD) for individual PFAS was defined as three times the standard deviation of the blank for those PFAS appearing in blanks; for all others, the LOD was estimated as the concentration producing a signal-to-noise ratio above three, based on analysis of the lowest calibration standard. The LODs ranged from 3.4- 197.9 pg/g (Table S 3).

**Total Oxidizable Precursor Assay Procedure**

Approximately 40 mg of each sample were weighed in a 50 mL falcon tube to which were added 30 mL of MilliQ water, 0.48 g of potassium persulfate and 0.456 mL of NaOH (10 M). The tubes were placed in a temperature-controlled oven at 85⁰C for 6 hours. After cooling samples were amended with 50 μl of a 200 pg/μl solution of isotopically labelled standards and their pH was adjusted between 5-9 using HCl (33%). Solid phase extraction (SPE) was carried out using Oasis WAX SPE cartridges (150 mg, 6 mL, Waters). Cartridges were conditioned with 4 mL each of 0.1% NH₄OH in methanol, methanol and water after which samples were loaded and extracted under vacuum, followed by a rinse with 4 mL of MilliQ water. After drying, samples were eluted into new 13 mL tubes with 4 mL each of 0.1% NH₄OH in methanol and methanol. Samples were dried to approximately 1 mL and 50 μl of a 200 pg/μl solution of isotopically labelled standards were added. An aliquot of 250 μL of sample was combined with 250 μL of 4 mM NH₄OAc in water for LC-MS/MS analysis.

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1 Powley CR, George SW, Russell MH, Hoke RA, Buck RC. Polyfluorinated chemicals in a spatially and temporally integrated food web in the Western Arctic. Chemosphere. 2008;70(4):664-672.
Inventory Calculations

The total quantity of fluorine from all artificial turfs (ATs) in Stockholm was estimated using the measured TF concentrations in backing, blades, and fill. Weight-based TF concentrations (i.e. µg F/g) in backing were first converted to area-based concentrations (i.e. µg F/cm²) using a weight-to-surface-area conversion factor (1g/cm²) which was approximately the same for all backing samples. The resulting concentrations were multiplied by the area of the field to obtain the mass of TF per field from backing. For fill, weight-based concentrations (i.e. µg F/g) were multiplied by the estimated quantity of fill added to a field per year (which is estimated between 2 and 3 metric tons, we therefore used 2500 kg for these calculations), only one application of fill for each field was considered in the estimation calculation. For blades, weight-based concentrations (i.e. µg F/g) were converted to field area-based concentrations (µg F/cm²) using a conversion factor (0.01g/blade) and the density of the blades on the turf (~10 blades/cm²); thereafter, concentrations were multiplied by the area of the field to obtain the total quantity of fluorine from all of the blades in a single field. Finally, the amount of fluorine in backing, fill and blades of a given field were summed together to obtain the total amount of fluorine for that field. As an example calculation, Gröndals BP, which has a surface area of 4230 m², was found to have TF concentrations of 31984 ng F/g in backing:

\[
F(\text{Gröndals} \text{backing}) = 31\,984 \frac{ng\,F}{g} \cdot 1\,g/cm^2 \cdot 4\,230\,m^2 = 1.35\,kg\,F
\]

The TF concentration in filling was 136018 ng F/g:

\[
F(\text{Gröndals} \text{filling}) = 136\,018 \frac{ng\,F}{g} \cdot 2500\,kg = 0.34\,kg\,F
\]

And 31984 ng F/g in blades

\[
F(\text{Gröndals} \text{blades}) = 64\,690 \frac{ng\,F}{g} \cdot 100\,000 \frac{blades}{m^2} \cdot 0.01 \frac{g}{blade} \cdot 4\,230\,m^2 = 0.2736\,kg\,F
\]

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2 Magnusson K, Eliasson K, Fråne A, et al. IVL-report C 183 Swedish sources and pathways for microplastics to the marine environment: a review of existing data. Swedish Environmental Protection Agency. Published online 2017. Accessed April 14, 2022.
The total amount of Fluorine for Gröndals is therefore estimated as:

$$F(Gröndals_{\text{total}}) = F(Gröndals_{\text{backing}}) + F(Gröndals_{\text{filling}}) + F(Gröndals_{\text{blades}})$$

$$= 1.35 \ kg \ F + 0.34 \ kg \ F + 0.2736 \ kg = 1.967 \ kg$$

Details of the TF measured for the other fields can be found in Table S5.

The sum of the total fluorine found in the sampled fields is 57.387 kg. The lowest amount of fluorine was found in Kungsholmens Gym BP at 0.315 kg, while the highest was found at Kälvesta BP at 17.439 kg. These values were used as upper and lower bounds estimates for the ATs not sampled and were multiplied by 86 (the remaining fields in Stockholm that were not sampled). The sum total of these estimates plus the measured values provided a total estimate of the quantity of fluorine in ATs in Stockholm, resulting in a range spanning from 84.45 kg to 1557.16 kg.

**Extraction of Fluoride in Water**

Contributions from fluoride towards TF measurements were ruled out based on the negligible concentrations of fluorine in water extracts from a subset of three samples of backing, blades, and filling containing high TF (backing from Hammarby IP, filling from Rågsveds BP and blades from Stadhagens IP). They were extracted twice with MilliQ water (7 and 6 mL) using an ultrasonic bath. The combined extracts were concentrated under nitrogen in a heated bath (60 °C) to ca 200 µL, which were then analysed by CIC. Results showed an average of less than 760 ng F/g, much lower than the TF values for these samples, while the same samples spiked with 5000 ng F⁻ showed recovery of fluoride in water extract of 65% (see Table S3). However, we cannot rule out contributions from other inorganic fluorine species which may occur in the turf and were not extractable in water. Overall, these results point towards a polymeric organofluorine consistent with patent literature.
Table S1 - Fields selected for sampling, Vasaparken was not sampled because the field had been turned into an ice-skating rink. In Hammarby IP and Knutby BP, sand was listed as the filling, but an unknown filling material was observed in addition to sand. The backing differed between fields as well, with some being thicker than others. In general, the new fields had thinner backing than the old fields but no inventory information was supplied regarding the nature of the backing material.

| Field                  | Year of Installation | Filling Material   | Blades Type |
|------------------------|----------------------|--------------------|-------------|
| 1 VASAPARKEN           | 2008                 | EPDM               | MONO        |
| 2 GRÖNDALS BP          | 2008                 | EPDM               | MONO        |
| 3 RÅGSVEDS BP          | 2015                 | EPDM               | MONO        |
| 4 HÄGERSTENSÅSENS BP   | 2015                 | EPDM               | MONO        |
| 5 KAKNÄS BP            | 2019                 | EPDM               | MONO        |
| 6 HJORTHAGENS IP       | 2019                 | EPDM               | MONO        |
| 7 HAMMARBY IP          | 2012                 | Sand+Unknown       | MONO        |
| 8 KNUTBY BP            | 2018                 | Sand+Unknown       | MONO        |
| 9 STADSHAGENS IP B     | 2011                 | SBR                | MONO        |
| 10 SOFIA BP            | 2013                 | SBR                | MONO        |
| 11 MÅLARHÖJDENS IP     | 2008                 | TPE                | MONO        |
| 12 KÄRRTORPS IP        | 2009                 | TPE                | MONO        |
| 13 TANTO BP            | 2019                 | TPE                | MONO        |
| 14 KALVESTA BP         | 2019                 | TPE                | MONO        |
| 15 HASSELANGENS BP     | 2017                 | TPO                | MONO        |
| 16 VÄLLINGBY BP        | 2017                 | TPO                | MONO        |
| 17 KUNGSHOLMENS GYMN BP| 2017                 | Organic Material   | MONO        |
| 18 NORRA REALS BP      | 2017                 | Organic Material   | Duo Shape   |

**EPDM:** ethylene propylene diene monomer; **SBR:** styrene butadiene rubber; **TPE:** thermoplastic elastomer; **TPO:** thermoplastic olefin; **MONO:** thick blades that stand upright; **Duo Shape:** a combination of MONO blades and slit-film blades, which are thinner blades obtained by cutting a thin sheet of plastic.
Table S 2 - PFAS targeted in this study (natives, internal standards and recovery standards) with MS parameters for quantification and limit of quantification, L-EtFOSAA was only targeted for analysis following total oxidizable precursor assay

| Compound Name                              | Abbreviation | Precursor Ion | Quantitative Product Ion | Qualitative product ion | Internal Standard | IS transition | Native used for quantification | LOD (pg/g) |
|--------------------------------------------|--------------|---------------|--------------------------|-------------------------|-------------------|---------------|--------------------------------|-------------|
| Perfluorobutanoic acid                     | PFBBA        | 213           | 169                      |                         |                   | 313>C2-PFBBA   | 217>172 PFBBA                  | 163         |
| Perfluoropentanoic acid                    | PFPeA        | 263           | 219                      | 169                     | 313>C2-PFPeA      | 266>223 PFPeA |                              | 81.0        |
| Perfluorohexanoic acid                     | PFHxA        | 313           | 269                      | 119                     | 313>C2-PFHxA      | 315>270 PFHxA |                              | 35.0        |
| Perfluorohexanoic acid (linear)            | L-PFOA       | 413           | 169                      | 369                     | 313>C2-PFOA       | 417>372 L-PFOA |                              | 43.0        |
| Perfluorooctanoic acid (branched)          | Br-PFOA      | 413           | 169                      | 369                     | 313>C2-PFOA       | 417>372 L-PFOA |                              | 43.0        |
| Perfluorooctanoic acid (linear)            | L-PFOA       | 413           | 169                      | 369                     | 313>C2-PFOA       | 417>372 L-PFOA |                              | 43.0        |
| Perfluorononanoic acid                     | PFNA         | 463           | 419                      | 219                     | 313>C2-PFNA       | 468>423 PFNA  |                              | 197.9       |
| Perfluorodecanoic acid                     | PFDA         | 513           | 469                      | 269                     | 313>C2-PFDA       | 515>470 PFDA |                              | 79.5        |
| Perfluoroundecanoic acid                   | PFUnDA       | 563           | 519                      | 269                     | 313>C2-PFUnDA     | 565>520 PFUnDA|                              | 140.9       |
| Perfluorotridecanoic acid                  | PFDoDA       | 613           | 569                      | 169                     | 313>C2-PFDoDA     | 616>570 PFDoDA|                              | 54.0        |
| Perfluorotetradecanoic acid                | PFTriDA      | 663           | 619                      | 169                     | 313>C2-PFDoDA     | 616>570 PFTriDA|                              | 135.9       |
| Perfluoropentadecanoic acid                | PFPeDA       | 763           | 719                      | 169                     | 313>C2-PFPeDA     | 616>570 PFTriDA|                              | 51.0        |
| Perfluorobutanesulfonic acid               | PFBS         | 299           | 80                       | 99                      | 313>C2-PFBS       | 403>84 PFBS   |                              | 53.0        |
| Perfluorohexanesulfonic acid (linear)      | L-PFHxS      | 399           | 80                       | 99                      | 313>C2-PFHxS      | 403>84 L-PFHxS|                              | 40.0        |
| Perfluorohexanesulfonic acid (branched)     | B-PFHxS      | 399           | 80                       | 99                      | 313>C2-PFHxS      | 403>84 L-PFHxS|                              | 40.0        |
| Perfluorodecanesulfonic acid (linear)      | L-PFOS       | 499           | 80                       | 99                      | 313>C2-PFOS       | 503>80 L-PFOS  |                              | 137.0       |
| Perfluorodecanesulfonic acid (branched)     | B-PFOS       | 499           | 80                       | 99                      | 313>C2-PFOS       | 503>80 L-PFOS  |                              | 43.0        |
| Perfluorodecanesulfonic acid (linear)      | L-PFDS       | 599           | 80                       | 99                      | 313>C2-PFOS       | 503>80 L-PFDS  |                              | 46.0        |
| Perfluorodecanesulfonic acid (branched)     | B-PFDS       | 599           | 80                       | 99                      | 313>C2-PFOS       | 503>80 L-PFDS  |                              | 46.0        |
| Perfluorooctanesulfonamide (linear)        | L-PFOSA      | 498           | 78                       | 478                     | 313>C2-PFOSA      | 506>78 L-PFOSA |                              | 42.0        |
| Perfluorooctanesulfonamide (branched)      | B-PFOSA      | 498           | 78                       | 478                     | 313>C2-PFOSA      | 503>80 L-PFOSA |                              | 42.0        |
| N-Ethyl perfluorooctanesulfonamidoacetic acid | L-EtFOSAA   | 584           | 419                      | 526                     |                  | 589>419 L-EtFOSAA|                              | 3.40        |
Table S 3 – Details of extraction of fluoride in water and TF concentrations (ng F/g) for comparison.

| Sample                                      | Spike (ng) | Measured F (ng; blank subtracted) | Concentration (ng F/g) | Recovery of F (%) | TF (ng F/g) |
|---------------------------------------------|------------|-------------------------------------|------------------------|-------------------|-------------|
| Hammarby IP (backing)                       | <LOD       | <LOD                                |                        |                   | 244831      |
| Rågsveds BP (filling)                       | 3.21       | 37.32                               |                        |                   | 309531      |
| Stadhagens IP (blades)                      | 90.53      | 2240.77                             |                        |                   | 660953      |
| Hammarby IP (backing) + F                   | 5000       | 2828.19                             | 82777.22               | 58.45             |             |
| Rågsveds BP (filling) + F                   | 5000       | 3416.75                             | 196402.2               | 68.35             |             |
| Stadhagens IP (blades) + F                  | 5000       | 3385.59                             | 40821.35               | 65.84             |             |

LOD: limit of detection
| Field                        | Backing |              | Filling |              | Blades |
|------------------------------|---------|--------------|---------|--------------|--------|
|                              | PFAS ng F/g | EOF ng/g | TF ng/g | PFAS ng F/g | EOF ng/g | TF ng/g | PFAS ng F/g | EOF ng/g | TF ng/g |
| Gröndals BP                  | 0.225   | <LOD        | 31984   | <LOD        | 163.2   | 136018  | <LOD        | 192.1   | 64690  |
| Hammarby IP                  | <LOD    | 32.8        | 244831  | <LOD        | 171.1   | 34421   | <LOD        | <LOD    | 75249  |
| Hjorthagens IP               | 0.293   | 23.3        | 312986  | 0.150       | <LOD    | 36210   | <LOD        | 65.3    | 71406  |
| Hägerstensåsen IP            | 0.047   | <LOD        | 16480   | <LOD        | <LOD    | 248792  | <LOD        | 33.6    | 44670  |
| Hässälängens BP             | <LOD    | <LOD        | 23437   | <LOD        | 26.2    | 166842  | <LOD        | <LOD    | 39827  |
| Kaknäs BP                    | 0.066   | <LOD        | 124370  | 0.039       | <LOD    | 203309  | <LOD        | 26.2    | 24284  |
| Knutby BP                    | 0.074   | <LOD        | 24713   | <LOD        | <LOD    | 96913   | <LOD        | <LOD    | 44617  |
| Kungsholmens Gym BP          | 0.095   | <LOD        | 28648   | <LOD        | <LOD    | 14238   | <LOD        | <LOD    | 23564  |
| Kälvesta BP                  | 0.140   | 63.6        | 243914  | <LOD        | 179.2   | 97490   | <LOD        | <LOD    | 80341  |
| Kärrtorps IP                 | <LOD    | <LOD        | 24778   | <LOD        | <LOD    | 201294  | <LOD        | 64.7    | 43840  |
| Mälarhöjdens IP              | 0.079   | <LOD        | 36022   | <LOD        | 143.3   | 173539  | <LOD        | 29.4    | 44367  |
| Norra Reals BP               | 0.056   | <LOD        | 25210   | <LOD        | 11909   | <LOD    | 11909      | <LOD    | 29073  |
| Rågsveds BP                  | <LOD    | 25.7        | 88310   | <LOD        | <LOD    | 309531  | <LOD        | 44.8    | 43088  |
| Sofia BP                     | 0.480   | 144.9       | 37159   | <LOD        | 45.2    | 43115   | <LOD        | <LOD    | 29356  |
| Stadshagens IP               | 0.089   | <LOD        | 42268   | 0.173       | <LOD    | 36183   | <LOD        | <LOD    | 660953 |
| Tanto BP                     | 0.782   | <LOD        | 32276   | <LOD        | 18439   | <LOD    | 18439      | <LOD    | 190717 |
| Vällingby BP                 | <LOD    | 22.9        | 25048   | <LOD        | <LOD    | 277112  | <LOD        | 31.1    | 41818  |
Table S 5 – Details of calculations estimating the total amount of Fluorine present in the sampled fields

| Field            | Size (m²) | Backing | Blades | Filling | Total |
|------------------|-----------|---------|--------|---------|-------|
|                  | TF (ng F/g) | TF (ng F/cm²) | F (kg) | TF (ng F/g) | Number of Blades | Weight of Blades (g) | F (kg) | TF (ng F/g) | F (kg) | F in Field (kg) |
| Gröndals BP      | 4230      | 31984   | 31984  | 1.353   | 6490     | 4.23E+08     | 4.23E+06 | 0.2736 | 136018 | 0.3400 | 1.967 |
| Hammarby IP      | 540       | 244831  | 244831 | 1.322   | 75249    | 5.40E+07     | 5.40E+05 | 0.0406 | 34421  | 0.0861 | 1.449 |
| Hjorthagens IP   | 800       | 312986  | 312986 | 2.504   | 71406    | 8.00E+07     | 8.00E+05 | 0.0571 | 36210  | 0.0905 | 2.652 |
| Hägerstensåsens IP | 2400  | 16480   | 16480  | 0.396   | 44670    | 2.40E+08     | 2.40E+06 | 0.1072 | 248792 | 0.6220 | 1.125 |
| Håsselängens BP | 2400      | 23437   | 23437  | 0.562   | 39827    | 2.40E+08     | 2.40E+06 | 0.0956 | 166842 | 0.4171 | 1.075 |
| Kaknäs BP        | 7140      | 124370  | 124370 | 8.880   | 24284    | 7.14E+08     | 7.14E+06 | 0.1734 | 203309 | 0.5083 | 9.562 |
| Knutby BP        | 6000      | 24713   | 24713  | 1.483   | 44617    | 6.00E+08     | 6.00E+06 | 0.2677 | 96913  | 0.2423 | 1.993 |
| Kungsholmens Gym BP | 900    | 28648   | 28648  | 0.258   | 23564    | 9.00E+07     | 9.00E+05 | 0.0212 | 14238  | 0.0356 | 0.315 |
| Kälvesta BP      | 6825      | 243914  | 243914 | 16.647  | 80341    | 6.83E+08     | 6.83E+06 | 0.5483 | 97490  | 0.2437 | 17.439 |
| Kärterps IP      | 6825      | 24778   | 24778  | 1.691   | 43840    | 6.83E+08     | 6.83E+06 | 0.2992 | 201294 | 0.5032 | 2.494 |
| Målarhöjdens IP  | 6825      | 36022   | 36022  | 2.458   | 44367    | 6.83E+08     | 6.83E+06 | 0.3028 | 173539 | 0.4338 | 3.195 |
| Norra Reals BP   | 1890      | 25210   | 25210  | 0.476   | 29073    | 1.89E+08     | 1.89E+06 | 0.0549 | 11909  | 0.0298 | 0.561 |
| Rägsveds BP      | 2400      | 88310   | 88310  | 2.119   | 43088    | 2.40E+08     | 2.40E+06 | 0.1034 | 309531 | 0.7738 | 2.997 |
| Sofia BP         | 2400      | 37159   | 37159  | 0.892   | 29356    | 2.40E+08     | 2.40E+06 | 0.0705 | 43115  | 0.1078 | 1.070 |
| Stadshagens IP   | 6000      | 42268   | 42268  | 2.536   | 660953   | 6.00E+08     | 6.00E+06 | 3.9657 | 36183  | 0.0905 | 6.592 |
| Tanto BP         | 800       | 32276   | 32276  | 0.258   | 190717   | 8.00E+07     | 8.00E+05 | 0.1526 | 18439  | 0.0461 | 0.457 |
| Vällingby BP     | 6000      | 25048   | 25048  | 1.503   | 41818    | 6.00E+08     | 6.00E+06 | 0.2509 | 277112 | 0.6928 | 2.447 |
Figure S1 - Overview of experimental approach. A stratified random sampling design was applied using two strata. In the first stratum, 2 fields for each filling type were selected while in the second stratum, 2 fields installed pre-2010 and 2 fields installed after 2017 were selected for filling types EPDM and TPE. For each location, samples of backing, blades and filling were collected. All samples were subjected to TF, EOF and targeted PFAS analyses. Smaller subsamples were selected for F⁻ and TOPA analyses.
Figure S 2 - Percent recovery ± RSD of native PFAS spiked onto two types of filling: organic material (n=3 spiked samples) and EPDM (n=2 spiked samples), as well as backing (n=2 spiked samples), and blades (n=2 spiked samples)
Figure S 3 - Results of TOPA performed on samples of backing (B), Blades (Bl) and filling (F). The lower $\Sigma$PFAS concentration after TOPA for some samples can be explained by more losses in the more complicated sample preparation for TOPA analysis.
Figure S 4 – Artificial Turf Fields in Stockholm, TF amounts of the 17 fields investigated are shown.