

DIN 38407-42:2011-03 (E)

German standard methods for the examination of water, waste water and sludge -
Jointly determinable substances (group F) - Part 42: Determination of selected
polyfluorinated compounds (PFC) in water - Method using high performance liquid
chromatography and mass spectrometric detection (HPLC/MS-MS) after solid-liquid
extraction (F 42)

Contents	Page
Introduction	5
1 Scope	6
2 Normative references	7
3 Terms and definitions	7
4 Principle	7
5 Interferences	8
5.1 General	8
5.2 Interferences encountered during extraction and processing of extracts	8
5.3 Interferences encountered during high performance liquid chromatography and mass spectrometry	8
6 Designation	9
7 Reagents	9
8 Apparatus	11
9 Sampling	12
10 Procedure	12
10.1 General	12
10.2 Sample preparation	12
10.3 Extraction	12
10.4 High performance liquid chromatography (HPLC)	13
10.5 Detection	14
10.6 Blank value measurements	14
11 Calibration	15
11.1 Principles	15
11.2 Calibration using an external standard	16
11.3 Calibration using an internal standard	17
11.4 Calibration check	18
12 Determination of recoveries	19
12.1 Recoveries of the method	19
12.2 Recovery rates of internal standards	20
13 Evaluation	21
13.1 General	21
13.2 Verification of individual substances	21
13.3 Calculation of the individual result	22

14	Expression of results	23
15	Test report	23
16	Performance data	23
Annex A (informative) Examples of sorbents, working conditions and recovery rates		27
A.1	Examples of sorbents and working conditions suitable for solid-phase extraction	27
Annex B (informative) Examples of suitable HPLC columns and chromatograms		34
B.1	Chromatographic conditions for the chromatogram shown in Figure B.1	34
B.2	Chromatographic conditions for the chromatogram shown in Figure B.2	35
B.3	Chromatographic conditions for the chromatogram shown in Figure B.3	36
B.4	Chromatographic conditions for the chromatogram shown in Figure B.4	37
B.5	Chromatographic conditions for the chromatogram shown in Figure B.5	38
Annex C (informative) Examples of selected diagnostic ions for identification and quantification ...		39
Annex D (informative) Examples for the expansion of the method		41
Annex E (informative) Explanatory notes		42
Bibliography		43
Figures Figure B.1 -- Chromatographic separation, example 1		34
Figure B.2 -- Chromatographic separation, example 2		35
Figure B.3 -- Chromatographic separation, example 3		36
Figure B.4 -- Chromatographic separation, example 4		37
Figure B.5 -- Chromatographic separation, example 5		38
Figure C.1 -- Example of a MS chromatogram of a surface water sample (extract shows branched and unbranched perfluorinated carboxylic acids)		40
Figure C.2 -- Example of a MS chromatogram of a surface water sample (extract shows branched and unbranched perfluorinated sulfonic acids)		40
Tables Table 1 -- Substances whose determination has been tested using this method		6
Table 2 -- Meaning of the indices		16
Table 3 -- Example of the assignment of internal standard substances to the analytes		17
Table 4 -- Performance data for HPLC/MS-MS measurement		24
Table 5 -- Performance data for drinking water		25
Table 6 -- Performance data for ground water		25
Table 7 -- Performance data for surface water		26
Table 8 -- Performance data for treated waste water		26
Table A.1 -- Examples of recovery rates - Ultra pure water (as in 7.2)		30
Table A.2 -- Examples of recovery rates - Drinking water		31

Table A.3 -- Examples of recovery rates - Surface water	32
Table A.4 -- Examples of recovery rates - Treated waste water (effluents)	33
Table C.1 -- Selected diagnostic ions (target compounds)	39
Table C.2 -- Selected diagnostic ions (internal standards)	39