Cyclic volatile methylsiloxanes (cVMS) are widely applied industrially produced chemicals. For example, cVMS are ingredients in personal care and household products. In addition, they are used as intermediates for silicon polymers. The general structure is a ring built of alternating oxygen and silicon atoms which are substituted with two methyl groups at each silicon atom. Due to their physical and chemical properties (e.g. high hydrophobicity, vapour pressures and Henry’s Law constants, relatively long modelled and measured half-lives), cVMS have the potential for long-range atmospheric transport, environmental persistence and bioaccumulation in food webs. It has been shown that cVMS octamethylcyclotetrasiloxane (D4), decamethyl-cyclopentasiloxane (D5) and dodecamethylcyclohexasiloxane (D6) are present in freshwater fish from several countries (e.g. Canada, Norway and Sweden). In 2018, D4, D5 and D6 were listed as Substances of Very High Concern under the EU REACH regulation. Hence, the target of this study was to investigate the occurrence and distribution of D4, D5 and D6 in fillets of German freshwater (bream) and marine (eelout) fish from major German rivers and the North and Baltic Sea, respectively. Samples of the German Environmental Specimen Bank (ESB) provide the opportunity to analyse time series. First, a solid/liquid extraction method with acetonitrile and hexane mixture was performed. Then, speciation and quantification of cVMS was carried out with a new GC-ICP-MS/MS coupling method. The application of the triple quadrupole technique allows the elimination of interferences, such as CO+ or N2+ ions, on the main silicon isotope (m/z=28) by the use of hydrogen as reaction gas. D5 was found in all samples from the riverine sampling sites. Furthermore, the results show that D5 concentrations were clearly higher than the observed D4 and D6 concentrations. In total, the highest cVMS burdens were determined in samples from the river Saar. In contrast, at ESB reference sampling site Lake Belau as well as in eelpout muscle tissue from the North and Baltic Sea no cVMS were found.