Most of the published studies focusing on the environmental and biological distribution of perfluorinated alkyl compounds (PFCs) have used triple quadrupole mass spectrometers for compound identification and quantitation. Multiple-reaction-monitoring (MRM) is a sensitive mass spectrometry technique that can be used on triple quadrupole instruments to provide trace level detection and monitoring. However, care must be taken when running complex matrices such as biological tissues, organic matter, and wastewater extracts since it is known that a number of compounds can share the same MRM transition as the target PFCs. As a result, using a single MRM transition may result in false positive data. To overcome this problem, a secondary MRM transition can be used to help confirm compound identification, but some of the PFCs do no produce useable secondary MRM transitions. Alternative mass spectrometry approaches that provide high sensitivity and more specificity than conventional MRM techniques are available, but they are rarely used at this time. A new type of mass spectrometer that combines ion trap capabilities with time-of-flight (IT-TOF) allows for the collection of full range accurate mass spectra in MS, MS/MS and MS<SUP>n</SUP> modes, obviating the need for monitoring discrete transitions. The sensitivity of this approach was found to be equivalent to that the MRM technique, and it allows rapid and ordered deconstruction of each of the target compounds to provide unambiguous identification. In this presentation, the first application of IT-TOF technology to the detection and quantification of PFCs in environmental and biological samples will be introduced. In addition, a new technique to identify unknown interfering compounds using the same technology will be discussed.