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Comprehensive two-dimensional gas chromatog. (GCxGC) was used to investigate the chem. compn. of the unresolved complex mixt. (UCM) of hydrocarbons in petroleum-contaminated marine sediments. The UCM hydrocarbons were extd. and sepd. with silica and silver-impregnated silica gel chromatog. to yield four fractions (branched alkanes and cycloalkanes, monoaroms., naphthalenes, and multiring PAHs) prior to GCxGC anal. GCxGC sepns. used a poly(di-Me siloxane) stationary phase for volatility selectivity on the first dimension and a 14% cyanopropylphenyl polysiloxane phase for polarity selectivity on the second dimension to fully resolve monoarom., naphthalene, and multi-ring PAH compds. from the UCM. A chiral γ -cyclodextrin phase was used for shape selectivity on the second GCxGC dimension to resolve individual branched alkanes and cycloalkanes in the sats. fraction of the UCM. The ability of GCxGC to resolve thousands of individual chem. components from the UCM will facilitate an understanding of the sources, weathering, and toxicity of UCM hydrocarbons.