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Black carbon determination in sediments and soils using a multi-elemental scanning thermal analysis (MESTA)

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The analytical problem of black carbon (BC) in the environment may not be solvable by a single method considering the complexity and heterogeneity of BC in the environment. But one thing is clear that we need some quantitative methods that are based on chemical nature, rather than the operational definition, to determine BC. By doing so, we know objectively what is assayed, its relevance to a BC question and its meaning when comparing results among methods. Recently, a multi-elemental scanning thermal analysis (MESTA) technique has been developed for the analysis of organic matter in complex matrices of solid and liquid samples. MESTA determines the thermal decomposition patterns of C, N and S in a sample simultaneously from ambient to 800 ^{o}C at a constant heating rate and given carrier gas composition. With the information of co-volatilized C, N and S at a temperature, MESTA provides multi-dimensional chemical signature for BC and the entire OC/BC continuum. Using MESTA to analyze BC, one does not have to force a predetermined criterion when the BC definition in question is not yet clear and more information is needed for the definition. We examine the application of MESTA to BC analysis of sediment and soil samples. BC reference materials including: hexane soot, CRM coal, NIST-SRM1649a urban dust, wood char, grass char, vertisol, mollisol, graphite and an active charcoal sample were used in this study. The results of MESTA reveal that whereas there is common chemical signature of BC in various samples, there are also differences. For example, activated charcoal, hexane soot and graphite all have condensed carbon volatilized at temperature higher than 550 ^{o}C (under 33 % oxygen and 50 ^{o}C/min. heating rate and the condition applies to all cases in this study). The volatilization temperatures were in an ascending order from activated charcoal to graphite. Most graphite carbon did not volatilized

below 800 ^{o}C while all carbon in other materials did below 700 ^{o}C. CRM coal has carbon volatilized between 500-600 ^{o}C but it has co-volatilized N as high as 3 %, on a weight basis, in that component and that may disqualify it as BC. Grass char has all carbon volatilized below 500 ^{o}C and may contain no BC according to MESTA. The volatilization temperature in a MESTA increases as the sample heating rate increases. We examined the optimal conditions of MESTA for BC analysis. The advantages and weakness of MESTA in BC analysis will be discussed. In most cases, the charring problem of MESTA was not serious. If it is, verification of that with a proper chemical method could solve the problem. We conclude that MESTA is a sensitive and quantitative alternative for BC analysis. It provides objective chemical signature of the OC/BC continuum in a sediment or soil sample.