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Techniques for VOC Soil Samples

The inherent problems of volatilization and biodegradation of soil samples has prompted the EPA regions and state agencies to adopt Method 5035 for sampling preservation and purging of soils for volatile organic analyses. The following is an overview.

Sampling - Take 3 soil samples using either EnCore[™] samplers (capping at once) OR preweighed vials (5-25 gm depending on site or state requirements), 1 vial preserved with methanol, the other 2 with bisulfate. Also, in both cases, fill a 2-4 ounce soil jar for dry weight determination. Combinations of part vial and part EnCore[™] sampling have been used and extra vials or EnCores[™]

may be needed for quality control purposes. Ask the laboratory for additional information.

Soil (unknown VOC level) Sampling EnCore™ Vial Option Option 1 vial with methanol 3 EnCores ™ 2 vials with stir bar & 1 soil jar bisulfate 1 soil jar Preservation Ice @ 4 ± 2° C ■ Lab must preserve Lab must analyze or analyze within 14 days within 48 hours of sampling of sampling Extrude into Extrude into 2 vials with stir 1 vial with methanol bar & bisulfate methanol vial **Analysis High Level** Low Level **Analysis Analysis** (Also used when low levels exceed 200 μg/Kg) Purge by gas Purge by agitation, displacement heat, & gas only displacement Analyze by GC or GC/MS

Final concentrations normalized to

dry weight basis

Preservation - Ice EnCores[™] and/or vials at 4+/- 2C and ship overnight to the lab. Within 48 hours the lab must extrude all EnCores[™] into vials and preserve with either methanol (1 vial for high level extraction and analysis) or with bisulfate solution (2 vials for level analysis) to act as a biocide. Magnetic stir bars present in the vials before addition of soil are needed later in the purging process. If highly alkaline soils are being preserved for low level analysis, checks for significant effervescence and buffering capacity should be written into the project plan and implemented. Contact the laboratory for additional instructions.

Analysis - Low Level - (less than 200 μ g/Kg) Analysis should start within 14 days of sample collection for bisulfate-preserved samples holding times for alkaline samples not preserved with bisulfate vary from state to state. Samples are placed in automated closed loop purge and trap systems where, unlike traditional gas displacement purging systems, the soil is also agitated with a stir bar. The entire vial is placed unopened in an autosampler and before purging commences, surrogates and internal standards are added without opening the vial. After purging, the trapped volatiles are desorbed and analyzed by either GC (8021) or GCMS (8260) Methods.

Analysis - High Level - (greater than 200 μ g/Kg) If the target compounds are present in concentrations well above 200 μ g/Kg (normalized to dry weight) a second purge is done using an aliquot from the methanol preserved vial.

The VOC's are purged in this case by gas displacement only (Method 5030) and then desorbed onto a GC or GCMS.

