

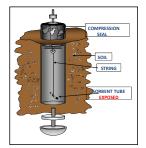
COLLECTION AND ANALYSIS OF PERFLUOROCARBON TRACER COMPOUNDS



Perfluorocarbon Tracers (PFT) Sample Collection and Analysis

Perfluorocarbon tracer (PFT) analysis is being used in support of the Carbon Capture, Utilization, and Storage (CCUS) of Carbon Dioxide. PFTs are added to the CO2 injection stream at the wellhead. Subsequent monitoring near the surface using sorbent tubes may indicate possible leakage from the reservoir.

Samples are collected using glass tubes filled with Ambersorb® adsorbent. A discreet volume of air is collected from the field site onto the sorbent. The tubes are sent to NETL, where they are analyzed utilizing thermal desorption with cryogenic focusing gas chromatography/mass spectrometry (GC/MS) with chemical ionization (CI) and selected ion monitoring (SIM). PFTs can be measured down to the concentration range of 200 femtoliter/liter of air, i.e. parts per quadrillion.



Detachable Head Penetrometer used for collecting tracer gas samples

LX,

Perfluorodimethylcyclobutane (PDCB)

Glass tubes filled with Ambersorb® carbonaceous polymer adsorbent



Perfluorotrimethylcyclohexane (PTCH)



Sample tubes collected in the field, are dried for at least 2 hours with ultra high purity helium.



Agilent® 6890N gas chromatograph (GC)
Gerstel® cryogenic focusing unit with autosampler
Agilent® 5975 mass selective detector (MSD)

Cryogenic Focusing Parameters

Cooled injection system (CIS) is cooled to -130 $^{\circ}\text{C}$ using liquid nitrogen.

Sample tube is automatically placed in thermal desorption unit (TDU)

TDU is heated to 350° C for 9 minutes to desorb PFCs onto injector. CIS is heated to 240° C to inject sample into the GC.

GC Parameters

GC Column : Varian CP-Sil 5 CB 100m X 0.32mm, 5.0 uM GC Oven temperature 35°C; Hold 22 minutes; Ramp 10°C to 150°C; Hold for 2 minutes.

Carrier Gas Flow: 1.2 ml/min.

MS Parameters

Chemical Ionization (CI) MS with selected ion monitoring (SIM) m/z 300; m/z 350; m/z 400; m/z 450.

 \mbox{CI} uses methane ($\mbox{CH}_4\mbox{)}\,$ as the make up gas.

CH₄ flow: 40 ml/min.

MS Source temperature = 150°C MS Quad temperature = 150°C Source Delay: 13 minutes

Example Chromatograms

