GC/MS Analysis for Identification of Unknown Organics

GC/MS analysis can be performed by IVA of hermetic devices, ampulization, static headspace, direct air injection (cylinders), or direct solvent injection (solvent extraction).

1) Analysis is performed at client requested temperatures.
    a. IVA of hermetic devices can be analyzed up to 200^{o}C. Customized bake times available.
    b. Ampulization up to 450^{o}C. Quartz ampules up to 1000^{o}C. Customized bake times available.
    c. Static Headspace up to 250-300^{o}C. (Recommend <250^{o}C). Bake times up to 999 minutes. Extended bake times optional.
    d. Direct Air Injection (cylinders) up to 150^{o}C.
    e. Direct Solvent Injection at ambient. Variety of solvents are available.
2) Standard Analysis Instrument Parameters
    a. Negative Electron Impact (EI)
    b. Helium carrier gas
    c. Scan range 35-550 amu.
        i. *Components with <35 mw will not be detected. (Methanol, Formaldehyde, etc).
        ii. **Will not identify inorganic acids, general inorganics, light atmospheric gases (H2, N2, O2, H2O)
        iii. ***If mw <35 is necessary (H2O, Methanol, gases, etc) should run standard IVA. If organic unknowns, run GC/MS. If both, should run both GC/MS and IVA for the "Full Picture".
        iv. PPB range detection limits.
3) Standard Report Format
    a. Identification of the top 10 unknowns by concentration
    b. Semi-quantitative results (non-calibrated)
        i. Results reported in units based on Relative %.
        ii. Relative % is compared relative to the other components detected.
    c. Narrative of analysis
        i. Introduction to describe the analysis and purpose
        ii. Description of any sample prep
        iii. Relevant Instrument parameters
        iv. Discussion of results

d. Photos of samples as received

e. Tentatively Identified Compound (TIC) summary

i. Sample info

ii. List of unknowns including retention time, CAS #, Tentative ID, Relative concentration, and Q-value (quality of match to NIST '11 Mass Spectral Library of > 243000 spectra)

f. Chromatograms of Control Blank and samples

g. Overlays of chromatograms illustrating relevant information (detected components, discrepancies between lots of samples, etc)

h. Mass spectra when applicable

4) Custom Report Format

a. Quantitative results for specific compounds available.

i. Specific compound information is required.

ii. NIST traceable standards may be required for analysis.

iii. Each calibration is performed and charged independently. Final cost will be based on the standards required (single-point cal vs 5-point cal, etc).

b. > 10 Unknown identifications optional.

i. Analysis time depends on number of unknown identifications

c. Additional options available—call to discuss.

Standard Turnaround Time = 5-10 Business Days.

How to choose which analysis is right for you:

-Sample size is important—Headspace vials can hold only 40 mm x 11 mm sample. Ampules can hold only 4-5 cm x 11 mm. Special large ampules can hold 8cm x 17mm (cannot seal certain atmospheres in large ampules).

-Are you looking for Hydrogen, Oxygen, Methane, Methanol, Moisture, Nitrogen, "Corrosive Gases", Ammonia, Formaldehyde, etc? This can be found using IVA only. GC/MS will not see these.

-Are you looking for unknown organics with mw>35 (mw 35~45 are sometimes tricky too)? These can be found via GC/MS.

-Organics, gas varieties and low mw or moisture? Both IVA and GC/MS will be required.

-GC/MS cannot identify hydrocarbon mixtures [ex.) gasoline, mineral spirits, oils], but will identify the individual hydrocarbons in those mixtures.

- Static headspace analysis is performed in room air only. Ampulization may be performed using optional gas matrices (dry N_2, Argon, etc).

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-Ampules vs Headspace? Headspace analysis is the preferred method when the samples fit in the vials (11mm x 40mm size or less) and analysis temperature is <250^{o}C. If over 250^{o}<400^{o}, ampules should be used. If you require a bake temperature of ~250^{o}, technical discussion will be required to determine the best test option. Early boilers benefit chromatographically from ampule analysis due to cryofocus option (can't be used with headspace analysis).

-Headspace analysis vs. solvent extraction? This depends on what you are looking for. If an off-gassing analysis is required headspace analysis will be applicable. If "impurities" or "surface contamination" analysis is needed a technical discussion will be required to determine the best test methods. There are many factors to consider; solvent extraction may lose very volatile components in the solvent peak whereas headspace analysis may not drive off all surface contamination, especially heavier components, and may create off-gassing from the sample material itself at elevated temperatures. We will presume the sample materials will off-gas consistently and any discrepancies between samples will be the impurities or contamination. Full technical discussion is required prior to submission.

Equipment:

- Agilent 7697A Headspace Autosampler
    - 111 Sample capacity, Temperature range-ambient to 300^{o} C, Equilibration times up to 999 minutes
    - 12-Position oven allows multiple sample equilibration at the same time
    - <1% RSD between samples reproducibility
    - 10, 20, and 22 mL vial capacity
    - Automatic vial leak check before analysis
    - Sample probe purge between samples to minimize carryover
    - Capable of liquid or solid applications, ideal for off-gassing studies
- Agilent 7890A GC
    - Advanced EPC, Retention time locking
    - Split/Splitless Inlet
    - Variable speed oven cooling fan
    - Liquid Nitrogen cryofocus option detects as low as C2 to C20 hydrocarbons.
    - Customized inlet adapter allows analysis of ampulized samples and IVA on hermetic devices
    - Several analytical GC column options available to optimize detection of specific components
- Agilent 5975C Mass Spectrometer
    - Scan range from 1.6-1050 amu at up to 12500 u/sec (We usually scan 35-550)
    - SIM, Scan and synchronous SIM/Scan capability

- Triple Axis detector maximizes signal-to-noise and lowers detection limits
- Inert Ion Source and gold/quartz quadrupole
- Deconvolution software enables better library search peak purities
- NIST ’11 Mass Spectral Library with >243000 spectra