DET

innovations in chemical detection

at XR = AUD/USD=0.79

INNOVATIONS IN CHEMICAL DETECTION FOR GC

featuring electrically heated, catalytically active ceramic elements that convert selected chemical compounds into electrical current

INEXPENSIVELY CONVERT AN NPD TO TID-10 SELECTIVE DETECTION AND GREATLY EXPAND THE APPLICATIONS FOR THE SAME BASIC EQUIPMENT

(e.g., Petroleum & Biofuels, Foods & Beverages, Flavors & Fragrances, Explosives, Selected Pesticides & Drug Compounds, etc.)

DET's TID-10 CERAMIC ION SOURCE (price Aud495 converts Thermo and Agilent NPD equipment to the following modes of selective detection:

NPD to TID-10 THERMIONIC SURFACE IONIZATION DETECTION - replace the NP ion source (bead) with a TID-10 ion source, plumb in N_2 , Air, or a combination thereof through the 3 gas lines that normally supply H_2 , Air, and Makeup gas to the NPD - provides selective detection of Oxygenates, some Halogenates, and compounds containing Nitro, Pyrrole, Thiol, and certain other functional groups.

NPD to TID-10 CATALYTIC COMBUSTION IONIZATION DETECTION (CCID) - replace the NP ion source with a TID-10 ion source, provide an Oxygen containing detector gas environment by plumbing in O_2, Air, or N_2O, combined with or without N_2, through the 3 gas lines that normally supply H_2, Air, and Makeup gas to the NPD - provides selective detection of compounds containing chains of Methylene (CH_2) groups (e.g. Alkanes, Alkenes, FAMEs, Triglycerides) with negligible Aromatic or Cyclo-Hydrocarbon responses and with additional differentiation between saturated versus unsaturated Carbon bonded compounds through judicious adjustment of the O_2 concentration in the detector gas environment.

Visit DET at Pittcon 2015, Booth 1853

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IMPLEMENTING TID-10 or CCID DETECTION on a THERMO TRACE 1300 GC - existing Thermo NPD hardware is compatible with mounting all types of DET ceramic ion sources and Thermo's existing NPD electronics are the most optimum available for operating all modes of detection - simply install a TID-10 ion source and provide the appropriate detector gases.

IMPLEMENTING TID-10 or CCID DETECTION on an AGILENT 6890/7890 GC - existing Agilent NPD hardware is compatible with mounting all types of DET ceramic ion sources, and Agilent's existing NPD electronics can be used, although the responses will not be the most optimum - simply install a TID-10 ion source and provide the appropriate detector gas environment - if desired, a significant improvement in signal-to-noise can be achieved by heating and polarizing the ion source with a stand-alone DET Current Supply ($1870 USD) instead of the Agilent Bead Voltage control - DET Supply provides more stable Constant Current ion source heating and - 5 V to - 45 V polarization selections for optimum response in all detection modes.

IMPLEMENTING TID-10 or CCID DETECTION on OTHER BRAND GC MODELS - requires replacing the existing NPD structure with a DET retrofit NPD/TID/CCID/FID hardware structure that can accommodate mounting of all DET ceramic ion sources.

THERMO TRACE ULTRA GC - DET tower structure Aud2350 mounts easily onto Thermo NPD detector base and uses the existing temperature and pneumatics controls associated with that base - install a TID-10 ion source and provide the appropriate detector gas environment - existing Thermo NPD electronics provide optimum control for all modes of detection;

VARIAN/BRUKER GC - DET tower structure Aud2200 mounts easily onto the Varian/Bruker TSD detector base and uses the existing temperature and pneumatics controls associated with that base - install a TID-10 ion source and provide the appropriate detector gas environment - existing TSD electronics can be used, although the responses will not be the most optimum - if desired, significant improvement in signal-to-noise can be achieved by substituting a stand-alone DET Current Supply to power the ion source;

SRI INSTRUMENTS GC - DET tower structure Aud2500 mounts easily onto SRI's heatable FID/NPD base, and uses the temperature and pneumatics controls associated with that base - install a TID-10 ion source and provide the appropriate detector gas environment - SRI's existing FID or NPD electronics can be used to adequately power DET ion sources for all modes of detection.

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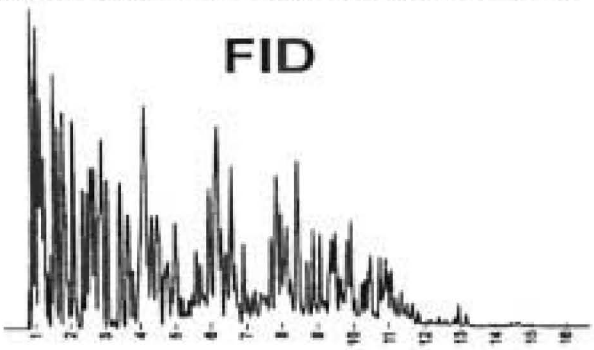
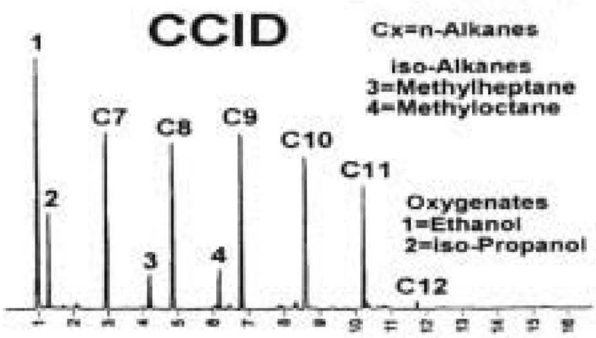
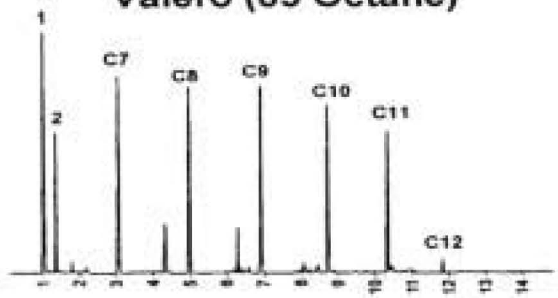
GASOLINE ANALYSIS



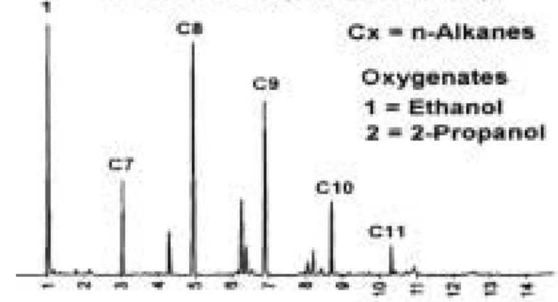


CCID ANALYSIS OF GASOLINES

Valero (85 Octane)

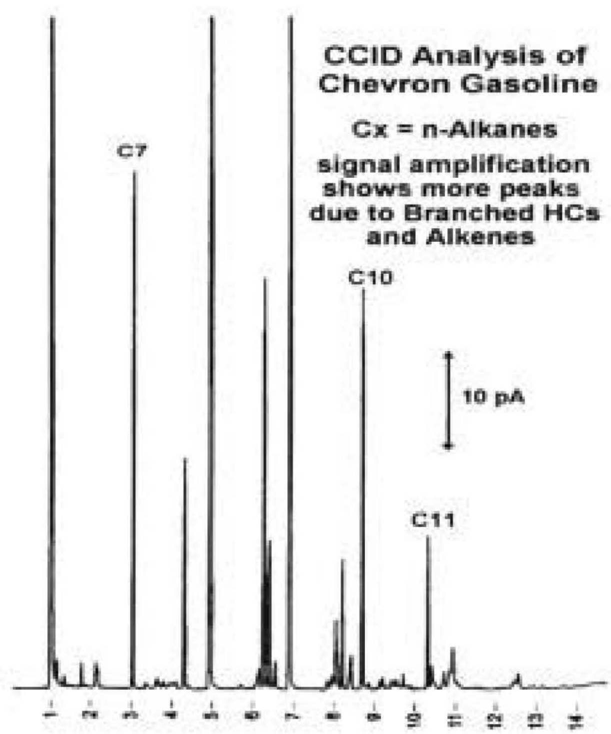


Chevron (87 Octane)



Catalytic Combustion Ionization (CCID) is a unique means of detecting n-Paraffin constituents in an otherwise complex petroleum matrix such as Gasoline. Other constituents such as Olefins and iso-Paraffins that contain chains of Methylene (CH_2) functional groups are also selectively detected while Aromatic and Naphthene compounds are not. The n-Paraffins dominate the detected peaks at low concentrations of Oxygen in the detector gas, while the relative response to iso-Paraffins and Olefins improves as the Oxygen concentration is increased.

The catalytically active TID-10 ceramic ion source used in CCID also responds to Oxygenated constituents via a Thermionic Surface Ionization process (TID) which is different than CCID. Hence, the gasoline chromatograms illustrated here are comprised of both CCID and TID selective responses. As shown above, these responses often differ according to which brand of Gasoline is analyzed.



Same chromatographic conditions were used for all data.
DET retrofit on Thermo Ultra GC, Air=30, O_2 makeup=50mL/min, TID-10 ion source heat=2.45A, polariz.= - 90V.
0.5µL gasoline, 30m x 0.53mm DB1, He=8, 40-60°C at 5°C/min, 60-190°C at 10°C/min, detector=250°C.

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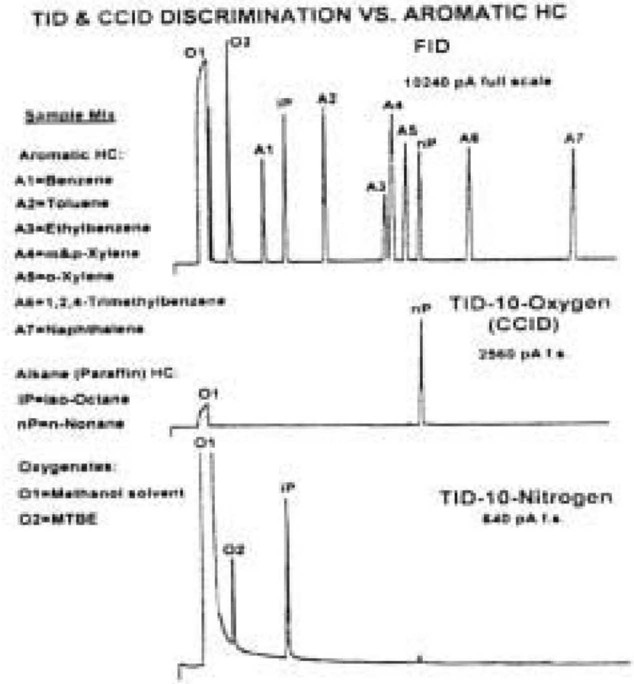
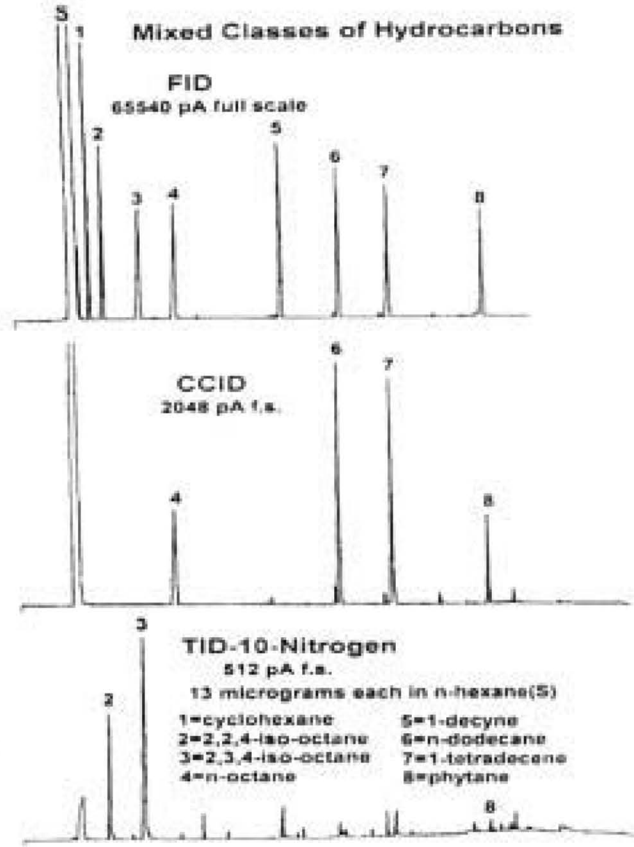
CATALYTIC COMBUSTION IONIZATION DETECTION (CCID)

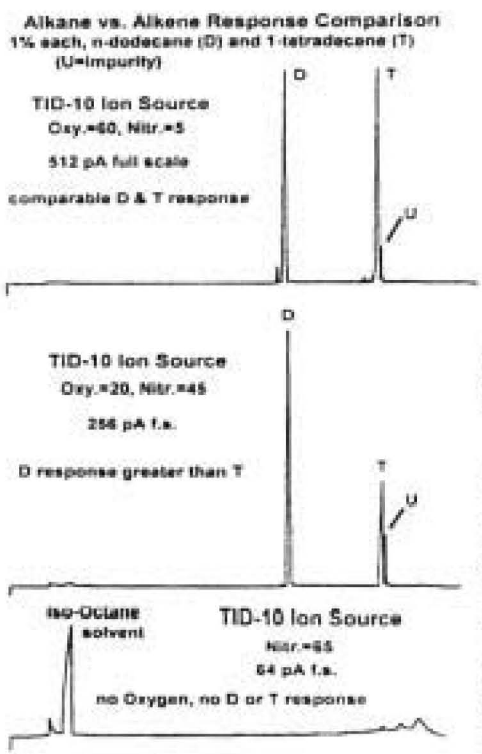
selective detection of Linear Chain Alkanes vs. other Hydrocarbons



CCID and TID DO NOT RESPOND to Aromatics (A1 - A7). CCID responds to Linear Alkane (nP), but not Branched Alkane (iP). TID responds to (iP) and Oxygenates.



CCID responds to multiple CH_2 groups in Linear Alkanes (4, 6), Alkene (7), and Branched Alkane (8), but NOT Alkyne (5), and NOT Branched Alkanes (2, 3) which have too few CH_2 groups.



CCID (TID-10 ion source in Oxygen gas environment)
Selective Detection of Methylene (CH_2) functional groups.

Depending on the Oxygen concentration, Linear Chain Alkanes with saturated Carbon bonds can be preferentially ionized versus Linear Alkenes with a Carbon double bond even though both classes of compounds may contain many CH_2 groups. Compounds with a Carbon triple bond (Alkynes) are similarly NOT very responsive to CCID detection even though they may contain many CH_2 groups. CCID selectivity dependence on compound bonding details provides unprecedented new chemical detection capability for GC.

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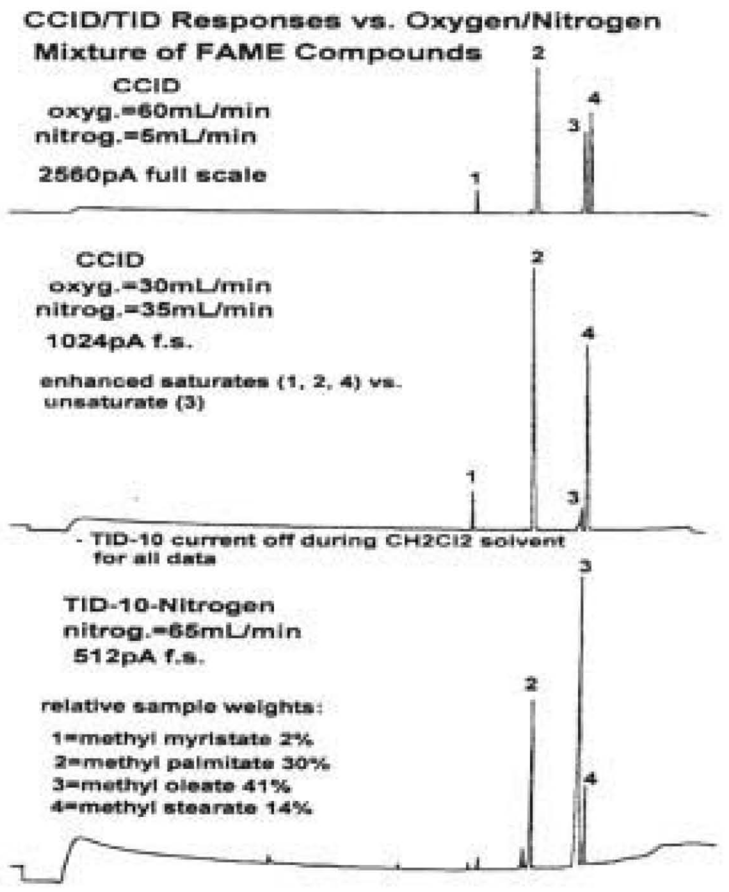
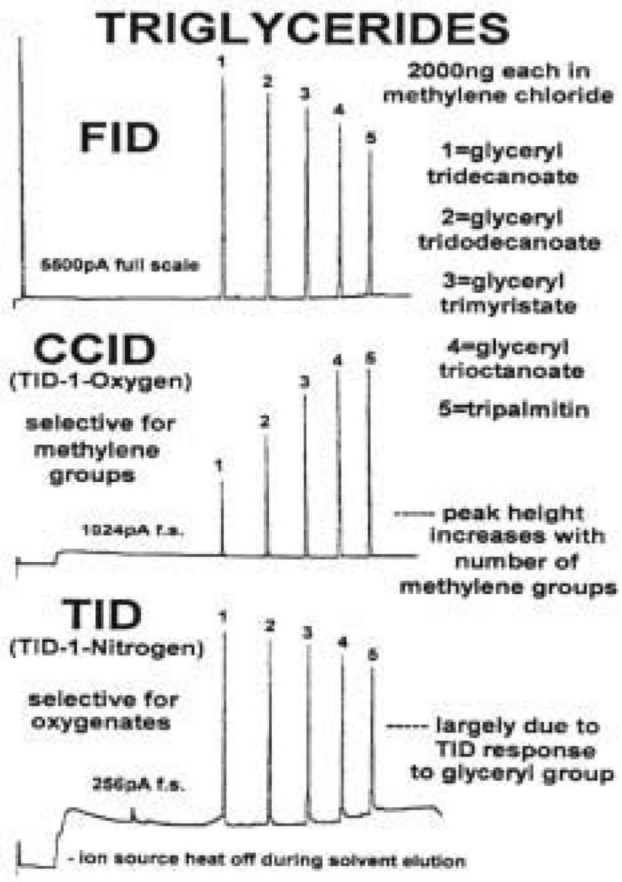
TID-10 (TID-1) & CCID SELECTIVE DETECTION TECHNOLOGY

FAMEs (Fatty Acid Methyl Esters) & TRIGLYCERIDES - compounds that contain chains of Methylene (CH_2) functional groups selectively detected by a CCID method, as well as Oxygen atoms selectively detected by a TID-10 (TID-1) method. (Note: TID-10 ion source is a recent more robust version of a TID-1 ion source.) TID-10 (TID-1) equipment is exactly the same as CCID equipment, and the only difference in the 2 detection methods is the concentration of Oxygen in the detector gases. CCID detects high concentrations of Hydrocarbon compounds, whereas TID-10 (TID-1) detects Oxygenated and some other Heteroatom compounds.





Analyses of a mix of saturated FAMEs (peaks 1, 2, 4) and an unsaturated FAME (peak 3). TID-10 responses with a Nitrogen gas environment qualitatively reflect the relative concentrations of the compounds. CCID response with a low Oxygen concentration preferentially detects the saturates vs. the unsaturated compound. Increasing the Oxygen, increases the CCID relative response of the unsaturated compound.

Analyses of a mix of saturated Triglyceride compounds. TID response is due to the Oxygen functionality of the compounds, and the relative peak heights are qualitatively similar to the FID response. In contrast CCID peak heights increase with increasing number of Methylene groups in the compounds.

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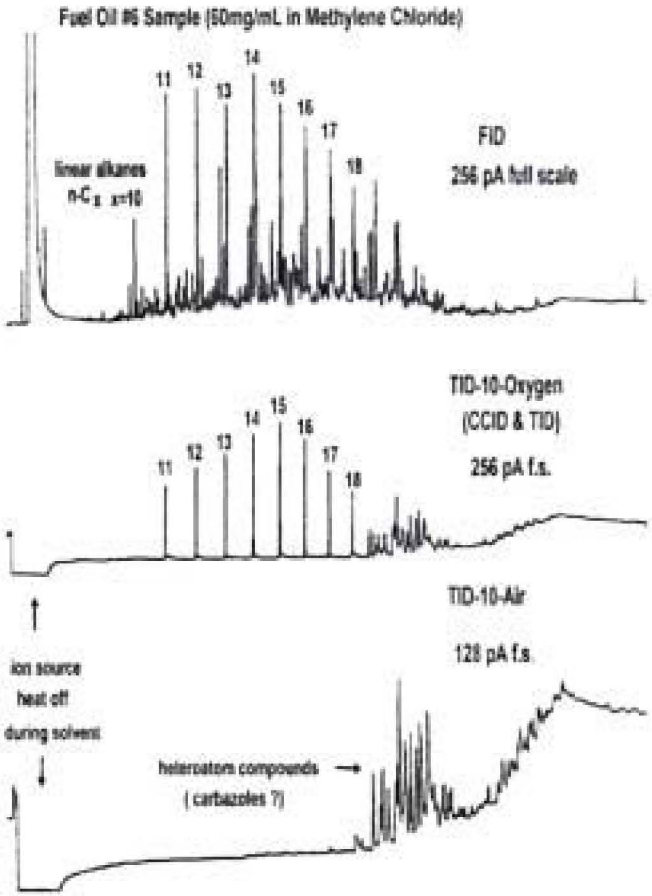
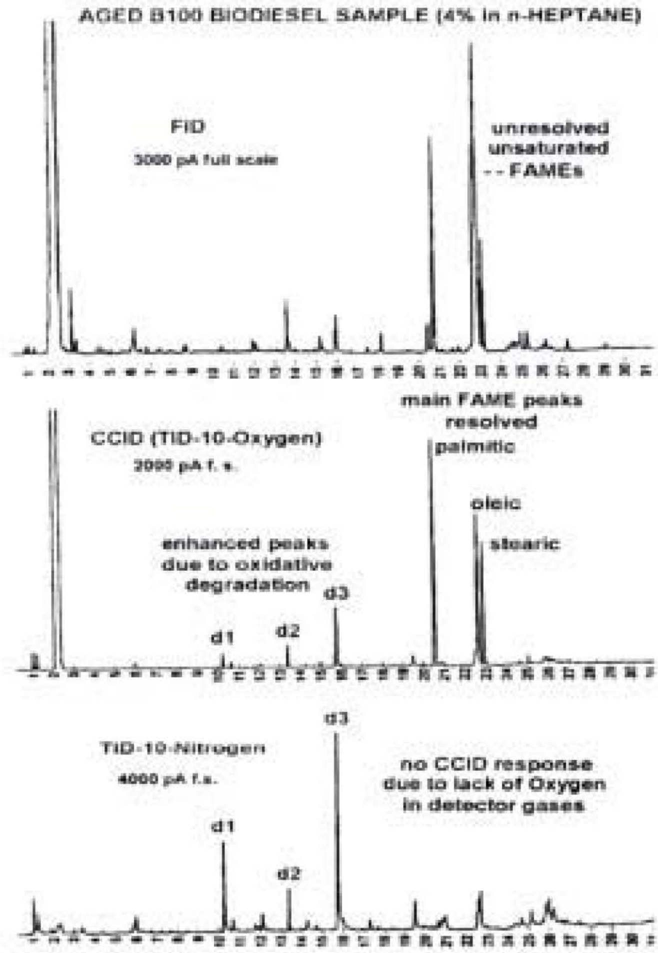
Example Chromatograms of PETROLEUM & BIOFUEL Samples
Unique TID-10 Thermionic Surface Ionization & Catalytic Combustion Ionization Profiles of Selected Constituents





TID-10 ion source in an Oxygen detector gas environment provided CCID selectivity for Linear Chain Alkanes versus other Hydrocarbon constituents of this oil sample. TID-10 Thermionic Surface Ionization in a detector gas environment with no or minimal Oxygen revealed selectivity for Indole and Carbazole type heteoatom compounds containing the Pyrrole functional group versus the Pyridine group.

Due to CCID response differences between saturated and unsaturated FAMEs, the CCID chromatogram provided resolved peaks for the 3 main FAME constituents of the sample compared to the poor resolution exhibited in the FID chromatogram. TID-10 detection with a Nitrogen gas environment provided selectivity and enhanced sensitivity for Oxygenated degradation compounds formed with aging. Sample aging in the presence of Air imparted a rancid aroma.

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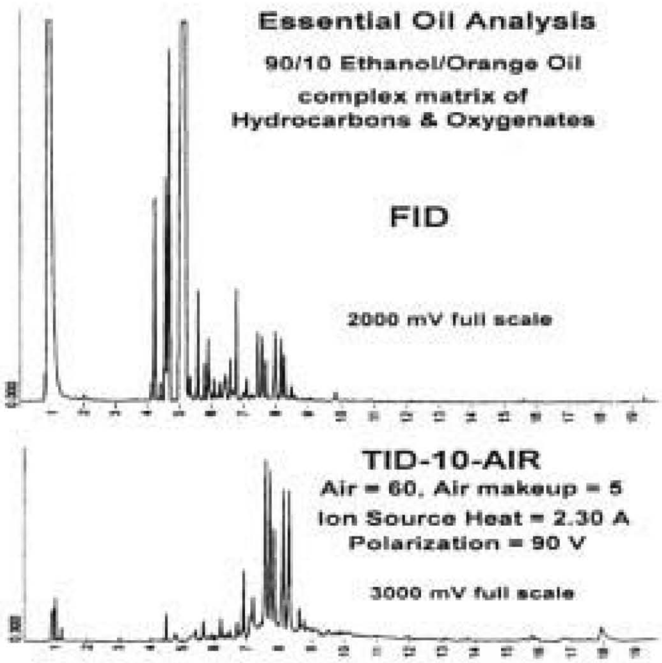
THERMIONIC SURFACE IONIZATION – SELECTIVE DETECTION OF OXYGENATES

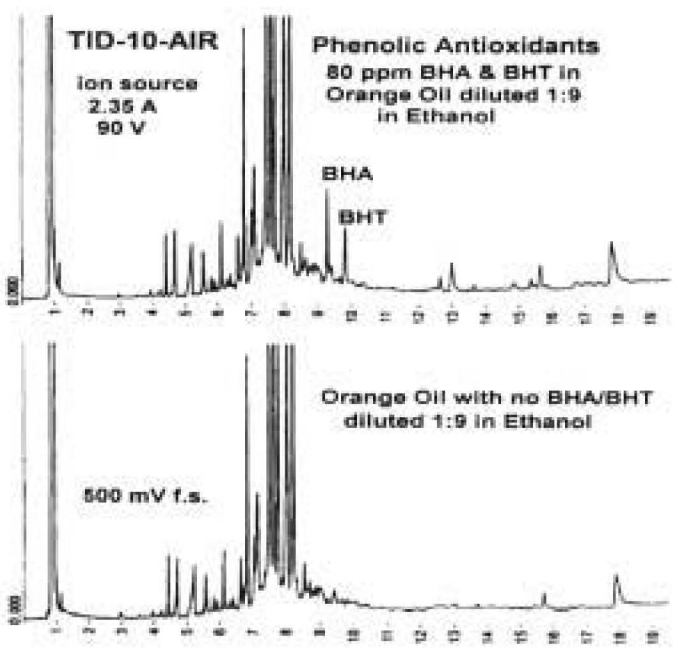


ESSENTIAL OILS

Essential Oils are complex mixtures of both Hydrocarbon and Oxygenated compounds, and an FID provides responses to all these compounds.

TID-10 ionization provides selective detection for the Oxygenated components with discrimination versus the Hydrocarbons. A detector gas environment of Air further suppresses responses for some classes of Oxygenates relative to other Oxygenate classes. Phenols, Glycols, and Carboxylic Acids are among those Oxygenates that remain very responsive in an Air environment. By contrast, Air suppresses responses due to Alcohols as illustrated by the very small Ethanol peak at the beginning of the TID-10-Air chromatogram.

Data were generated using a Thermo Trace Ultra GC retrofitted with DET NPD/TID hardware mounted on Thermo's NPD detector base, and operated using Thermo's NPD electronics. Air was supplied to the detector through the 3 gas lines that normally provide "H_2", Air, and Makeup gases to an NPD.



BHA and BHT are Phenolic Antioxidants that are widely used as preservatives in many food products. TID-10-Air detection provides enhanced responses for Phenol type compounds relative to other type constituents of Essential Oils.

Similar data can be achieved with simple adaptation of existing NPD equipment on the Thermo Trace 1300 GC. Optimum Oxygenate detection with Agilent NPD equipment is achieved by substituting a stand-alone DET Current Supply for Agilent's Bead Voltage as the power supply for a TID-10 ion source. For Varian/Bruker and SRI Instruments NPD GC models, optimum detection requires inexpensive DET Retrofit hardware and the DET Current Supply.

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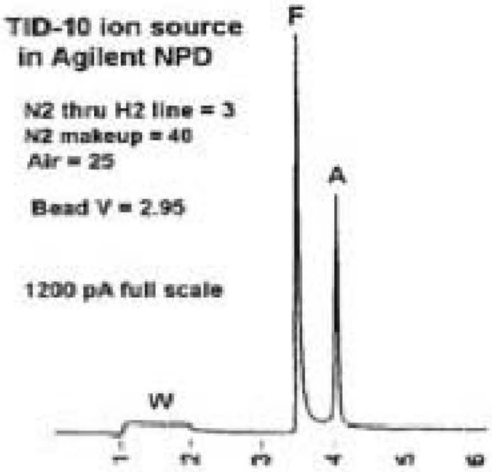
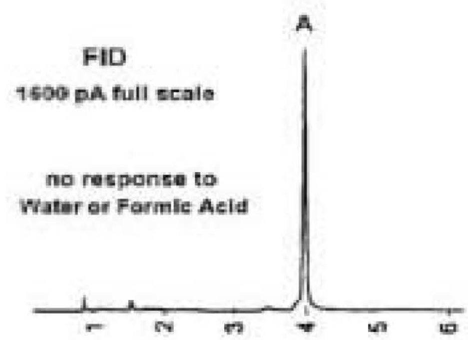
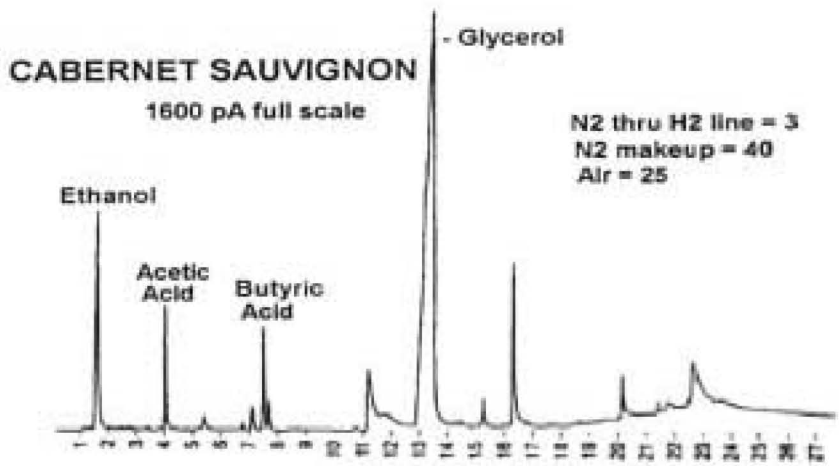
TID-10 Thermionic Surface Ionization Detection of Volatile Acids and other Oxygenated Compounds

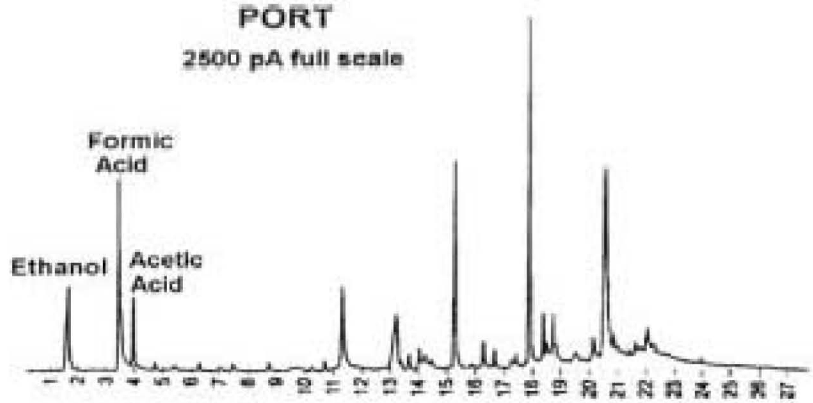
FORMIC (F) & ACETIC (A) ACIDS
0.1 % each in Water (W)





TID-10 Ion Source in Agilent NPD - Wines
0.17" ID collector insert, Bead V = 3.04





Selective detection of Oxygenates by means of the TID-10 Thermionic Surface Ionization process produces different magnitudes of response depending on the Oxygenate class. Volatile Acids, Phenols, and Glycols have especially large responses relative to other Oxygenates such as Alcohols. Vanillin and Methyl Salicylate are other examples high responding compounds, while Ethers typically have the lowest responses. The response to volatile acids includes Formic Acid which is not detected by an FID. TID-10 detection applied to complex samples such as Wine can provide a chromatographic fingerprint quite different than that of an FID. TID-10 responses using Agilent NPD Bead Voltage electronics can be improved with a smaller internal diameter insert placed inside the Agilent collector electrode to increase the magnitude of electric field for ion collection.

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DET RETROFIT NPD/TID/FID **HARDWARE TO FIT SRI GC MODELS. DET** equipment has an optimum concentric cylinder geometry that provides better stream-lined gas flow and efficient ion collection, and it is compatible with the same ion source mountings as used on **AGILENT GC** models.

COMPACT GC **ANALYZER** - portable size **SRI GC** modified with glass lined flash vaporization injector, **0.32** mm dia. fused silica column, **DET** detector hardware with ceramic ion sources, and **DET** Current **Supply** for precision ion source power.

DETector Engineering & Technology, inc.

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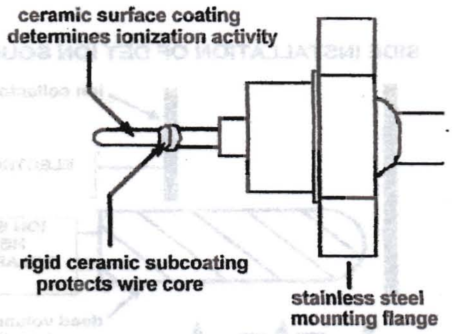
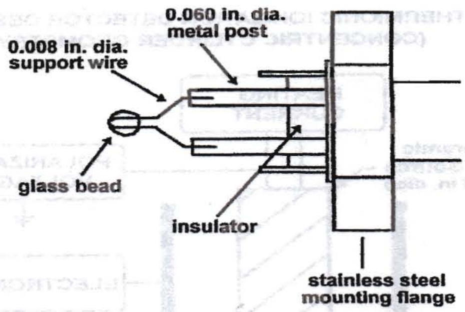
Australian Distributors; Importers & Manufacturers

NPD ION SOURCES (BEADS) USED IN AGILENT NPDS
COMPARISON OF CERAMIC VERSUS GLASS CONSTRUCTION



DET Ion Source Construction
(fits Agilent & DET structures)



Blos Bead for Agilent NPD

CERAMIC

1.) 1/16 inch diameter ceramic materials coated over an internal wire core.

2.) Ceramic ion sources remain a rigid solid surface at the 600 - 800°C temperatures required for NP detection.

3.) Additives in the ceramics withstand temperatures in excess of 1000°C so ion sources can be used with a wide variety of operating conditions without danger of melting.

4.) Ceramic sublayer coating completely covers the wire core of the ion source and protects it from corrosion from additives in the surface coating or from corrosive sample matrices. Sublayer additives are chosen to enhance the hardness and electrical conductance of that ceramic coating.

5.) Additives in the ceramic surface layer determine the ionizing and catalytic activity of the ion source. Many different surface coatings are possible to optimize NP response characteristics as well as provide other modes of thermionic ionization detection such as selectivity for Oxygenates and Halogenates.

6.) In operation, electrical resistance of the ion source wire core is 1.2 Ohms which ensures that heating power is mainly dissipated in the ion source rather than in the electrical lead wires and connectors.

7.) Physical size of the ion source minimizes sensitivity to gas flow changes in the detector.

GLASS

1.) Approximately 0.040 inch diameter glass bead deposited on the loop end of 0.008 inch diameter support wire.

2.) Glass bead is in a softened state at the temperatures required for NP detection. Microscopic examination of used beads often reveals internal bubbles and/or particulate growths on the surface. Some volatilization of bead material is also likely with vaporized material depositing on colder surfaces downstream.

3.) Detector operating conditions must be carefully controlled to avoid melting the glass.

4.) Thin support wire is easily bent out of shape by the slightest touch.

5.) Thin support wire is exposed to corrosion from additives in the glass as well as from corrosive sample matrices.

6.) Exposed support wire contributes some ionization in addition to the glass bead. Tailing of Phosphorus peaks is a characteristic of the NP responses.

7.) In operation, electrical resistance of the ion source is 0.19 Ohms which means that ion source heating power heats lead wires and connectors in addition to the ion source, and the temperature of the ion source is extremely sensitive to the stability of the heating power supply.

8.) Small physical size of the glass bead means the bead is very sensitive to fluctuations in gas flows, and positioning the bead relative to the incoming sample orifice is critical.