

CHEMICAL DETECTION PRODUCTS

by

DET

selected chemical compounds converted to electric current

by novel applications of the scientific principles of

THERMIONIC SURFACE IONIZATION (TID)

CATALYTIC COMBUSTION IONIZATION (CCID)

& FLAME IONIZATION (FID)

featuring a family of electrically heated ion sources

comprised of catalytically active ceramics

GAS CHROMATOGRAPH (GC) DETECTION

**Ceramic Ion Sources (beads) for NPD, TID, CCID
and Bare Wire Ignitor/Polarizer Probes for FID**

**Retrofit Detector Equipment for
Agilent, Thermo, Varian/Bruker, & SRI GCs**

STAND-ALONE TRANSDUCERS & MODULES

**screening/monitoring of selective chemicals in gas streams
including vapors generated by Thermal Desorption
or Thermal Oxidation of constituents in solids and liquids**

DETector Engineering & Technology, inc.

486 North Wiget Lane, Walnut Creek, California 94598-2408 USA

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innovations in chemical detection

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INTRODUCTION TO AN EXTRAORDINARILY VERSATILE CHEMICAL DETECTION TECHNOLOGY

The chemical detection methods described in this brochure evolved from an original design for an improved configuration of a Nitrogen-Phosphorus Detector (NPD) for Gas Chromatography (GC), and from a recognition that the NPD was just one member of a family of detectors that operate according to the principles of Thermionic Surface Ionization. The key components of this detection technology are an electrically heated, cylindrically shaped ion source element positioned on the axis of an ion collector cylinder. Ion source elements are comprised of DET's proprietary ceramic coatings applied over an inner wire core, and there is available a family of different coatings for converting different types of selected chemical compounds into detectable ion currents. There are unlimited possibilities for further varying the catalytic, ionizing, and semiconducting nature of the ceramics, so research and development of new formulations is an on-going process. These common components are used in all the different hardware implementations described in this brochure, such that the same equipment is readily adaptable from one mode of selective detection to another.

DET does not manufacture a GC instrument, so implementation of its detection methods requires adaptation of detector parts already existing on the GC. Specifically, FID and NPD detector bases on GC instruments are the most suitable because these type bases are heatable, and contain gas lines for supplying Hydrogen, Air, and usually Makeup gas in addition to the GC carrier gas. This means that FID/NPD temperature and pneumatics controls existing on the GC can be used for DET detection as well, although the detector gases may not necessarily always be Hydrogen and Air. For other type detector gases, it is usually a simple matter of connecting the appropriate gas to the "Hydrogen" and "Air" gas line inlets on the instrument.

Some GC manufacturers have detector hardware already designed to accommodate ion source elements manufactured by DET. For instruments that are not so designed, DET has developed detector tower structures that are easily mounted onto the existing FID/NPD base on that instrument, and then accommodate the DET ion sources. DET transducer hardware and ion sources are also available for non-GC screening and monitoring applications.

In most cases, the chemical detection methods described in this brochure are unique, and often provide sample responses and capabilities unavailable by other detection techniques.

Paul L. Patterson, Ph.D.

President

DET, inc.

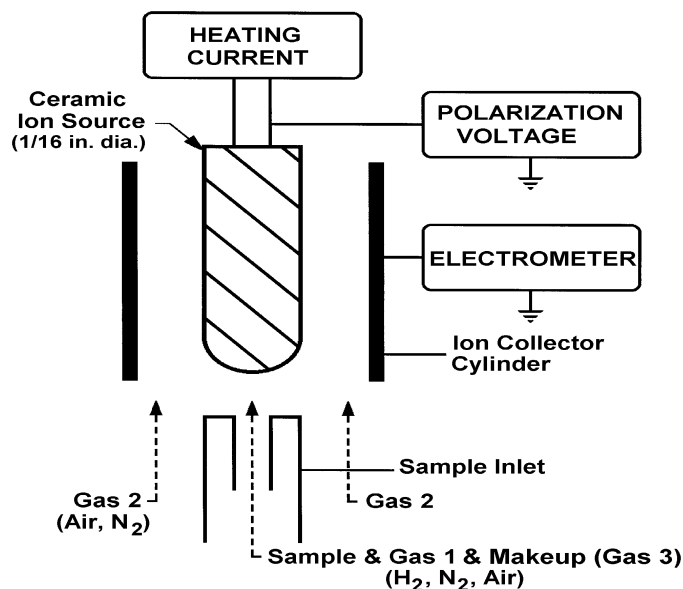
Thermionic Surface Ionization & Catalytic Combustion Ionization

Unique GC detection technologies convert selected chemical compounds into negative ion currents using electrically-heated ionizing surfaces made of catalytically-active ceramic materials.

OPTIMUM DETECTOR HARDWARE - concentric cylinder geometry used on the Agilent 6890/7890 NPD, Thermo Trace 1300 NPD, and all DET hardware features an ion source positioned on the axis of an ion collector, with top access for easy ion source changes.

OPTIMUM DETECTOR ELECTRONICS - Constant Current electrical heating of ion sources plus choice of ion source polarization ranging from - 5 Volts to at least - 45 Volts relative to the ion collector as available with Thermo NPD electronics or a stand-alone DET Current Supply . (existing Agilent, Varian/Bruker, SRI NPD electronics also work, but not most optimum)

THERMIONIC IONIZATION DETECTOR DESIGN
(CONCENTRIC CYLINDER GEOMETRY)



PARAMETERS THAT DETERMINE COMPOUND SELECTIVITY & SENSITIVITY:

1. catalytic ionizing activity of the ion source as determined by the composition of its ceramic coating (an unlimited number of ceramic formulations are possible);
2. temperature of the ion source (typically in the range 300 - 900°C);
3. composition of the gases flowing past the ion source (e.g., N₂, Air, O₂, N₂O, H₂, and combinations thereof);
4. the magnitude of the polarizing voltage between the ion source and collector.

Multiple modes of detection are achieved using the same basic equipment, a choice of different ion source ceramics, and various permutations of the 4 operating parameters - selectivity choices include compounds containing O, N, P, Cl, Br, I, Pb, Sn, or Si atoms, or NO₂, Pyrrole, CH₂, and certain other functional groups - new compound selectivities continue to be identified. Tandem configurations of different detection modes provide the ultimate versatility for simultaneous selective detection combinations.

Detection equipment is inexpensive, uncomplicated in design, easy to use, does not require ultrapure or exotic gases, and provides detection capabilities unmatched by any other type of GC detector technology.

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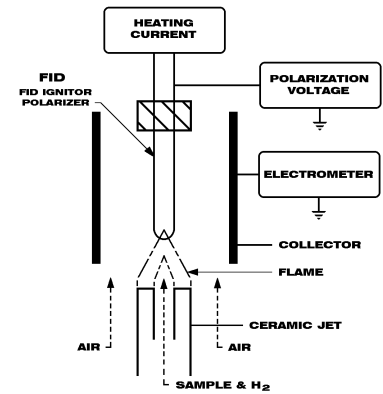
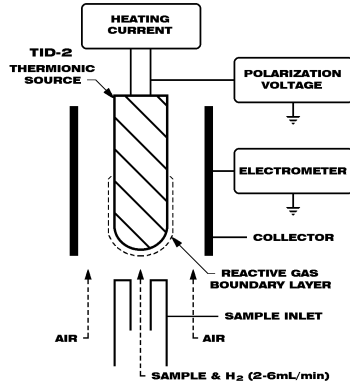
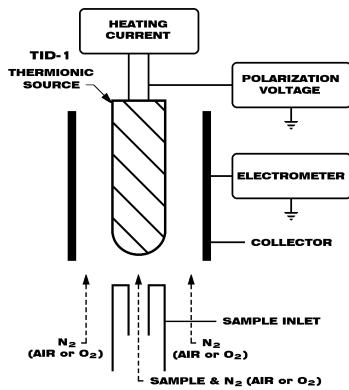
GC DETECTOR INNOVATIONS by DET

(different implementations of the same basic detector geometry)

TID/CCID (O, Cl, Br, NO₂, CH₂, etc.)

NPD (N, P)

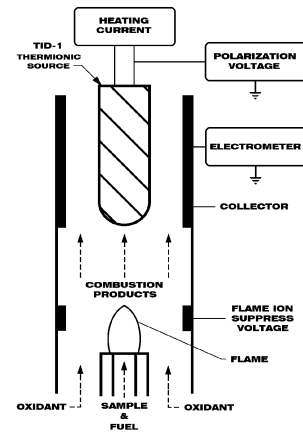
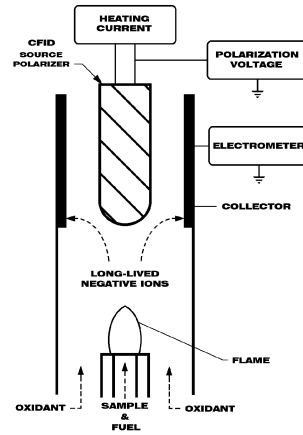
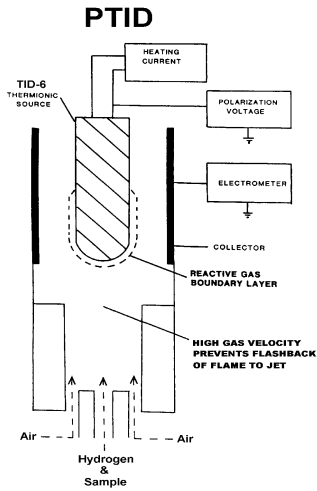
FID (universal)



PTID (P)

REMOTE FID (P, Pb, Sn, Si)

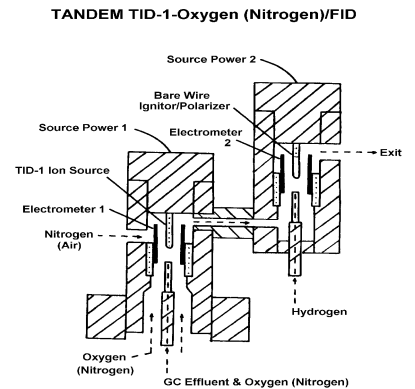
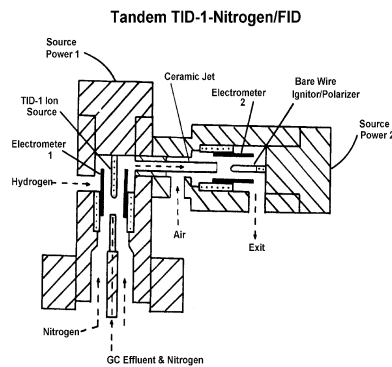
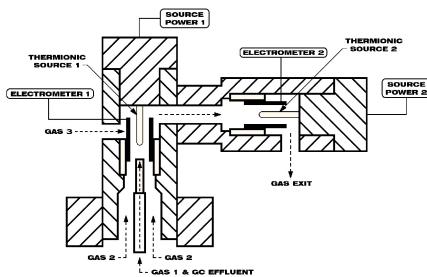
FTID (N, Cl, Br)



TANDEM TID

TANDEM TID-Nitrogen/FID

TANDEM TID-Oxygen/FID



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DET Chemical Detection Products based on Scientific Principles of THERMIONIC SURFACE IONIZATION and FLAME IONIZATION featuring Electrically Heated Ion Sources made of Proprietary Ceramics

multiple modes of selective detection using common detector components

COMMON COMPONENTS - inexpensive detection equipment features a concentric cylinder geometry in which interchangeable ion source elements are positioned on the axis of a collector electrode, with the ion sources heated by an electrical current and polarized at a negative voltage with respect to the collector - ion sources are small cylindrical rods formed from multiple layers of ceramic coatings molded over a wire core and mounted on a stainless steel flange.

THERMIONIC IONIZATION DETECTION (TID) - sample compounds directly impact the ion source and form gas phase ions by extracting negative charge from the ceramic surface - selectivity and sensitivity of detection depends on the catalytic ionizing activity of the ceramic, and on whether the detector gas environment is inert (e.g., Nitrogen) or oxidizing (e.g., Air or Oxygen) - available types of ceramic ion sources are as follows:

TID-10 - selective for some Nitro and Halogenated compounds at Femtogram and Picogram levels; many Oxygenates at Picogram and Nanogram levels with especially large responses for Phenols, Carboxylic Acids, Glycols, Glycerol, Vanillin, and Methyl Salicylate; Pyrrole vs. Pyridine functional groups; Water vapor at ppm levels with Air detector gas; also used in the Catalytic Combustion Ionization mode for selectivity to Methylene (CH₂) functional groups. (TID-10 has same response as earlier TID-1, but is more robust).

TID-3 - selective for volatile Halogenates such as Trihalomethanes.

TID-5 - selective for Br and I compounds with suppressed Cl response.

TID-7 - selective for Halogenates such as PCBs

NITROGEN PHOSPHORUS DETECTION (NPD) - NP selectivity turns on when the ion source is heated sufficiently (i.e., 600 - 800°C) to ignite a dilute mix of Hydrogen in Air to form a chemically reactive gaseous boundary layer around the hot ion source surface - samples are decomposed in the ignited boundary layer, and electronegative N and P decomposition products extract negative charge from the hot ion source to form detectable gas phase ions - DET has developed 2 types of ceramic ion sources for NP detection as follows:

TID-2 (Black Ceramic) - NP detection with negligible tailing of P peaks - 70fg P/sec detectivity;

TID-4 (White Ceramic) - NP detection with the largest possible N response - 70fg N/sec detectivity;

PHOSPHORUS THERMIONIC IONIZATION DETECTION (PTID) - a TID-6 ion source is located downstream of a small diameter flow restrictor such that when the ion source is heated sufficiently to ignite a pre-mixed stream of high concentration Hydrogen in Air, the high total gas flow prevents flame flash back from the source to the original mixing point of the Hydrogen and Air - like an NPD, the ignited chemistry remains as a boundary layer about the hot source - this mode provides selective detection for P compounds with very large signals and suppressed N response.

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REMOTE FID DETECTION (RFID) - a CFID ion source & collector electrode are located several centimeters downstream of a self-sustained Hydrogen/Methane/Air flame - ions produced by Hydrocarbon combustion dissipate rapidly downstream of the flame, but combustion of Lead (Pb), Tin (Sn), Phosphorus (P), or Silicon (Si) compounds produces long-lived ions that persist to be selectively detected at the downstream collector - an organic fueled flame improves selectivity. P response more uniform & selective than NPD, but not as sensitive.

FLAME THERMIONIC IONIZATION DETECTION (FTID) - an electrically heated TID-1 or TID-2 ion source located several centimeters downstream of a self-sustained Hydrogen/Methane/Air flame re-ionizes the electronegative neutral products of the flame combustion of samples - a TID-2 source provides selectivity for Halogenates, while a TID-1 source provides selectivity for Halogenates and Nitrogen compounds.

FLAME IONIZATION DETECTION (FID) - an FID Probe consisting of an uncoated bare wire loop, and a collector electrode are located adjacent a self-sustained Hydrogen/Air flame burning at an unpolarized ceramic tipped jet - the FID Probe serves as flame ignitor and polarizer, and the flame ionization provides Universal detection of organic compounds.

HOT WIRE COMBUSTION IONIZATION DETECTION (HWCID) - similar to a PTID configuration except a heated bare wire FID Probe is used to maintain an ignited Hydrogen/Air boundary layer - Universal detection like an FID, but sensitivity about 100 times less than an FID - does not require a jet structure - provides about a factor of 2 enhancement for Aromatics vs. Alkane Hydrocarbons.

CATALYTIC FLAME IONIZATION DETECTION (CFID) - uses a self-sustained flame similar to an FID except includes an electrically heated ceramic CFID ion source to augment the gaseous flame ionization with surface ionization from the hot catalytic ion source - Universal detection with similar response factors for Halogenates and Hydrocarbons.

CATALYTIC COMBUSTION IONIZATION DETECTION (CCID) - uses a catalytic TID-10 ion source heated to 300 - 400°C in an oxidizing gas environment to ignite a momentary burst of flame ionization as individual sample compounds elute from the GC column and impact the ion source - provides selective ionization of Methylene (CH₂) groups in linear chain Alkane, FAME, and Triglyceride compounds with discrimination vs. compounds with unsaturated Carbon double bonds, and no response to Aromatic or Cyclo-HCs.

TANDEM DETECTION (TID/NPD, NPD/TID, FID/FTID, TID/FID, etc.) - 2 simultaneous detector signals with many different possible combinations of ion sources and detector gas environments.

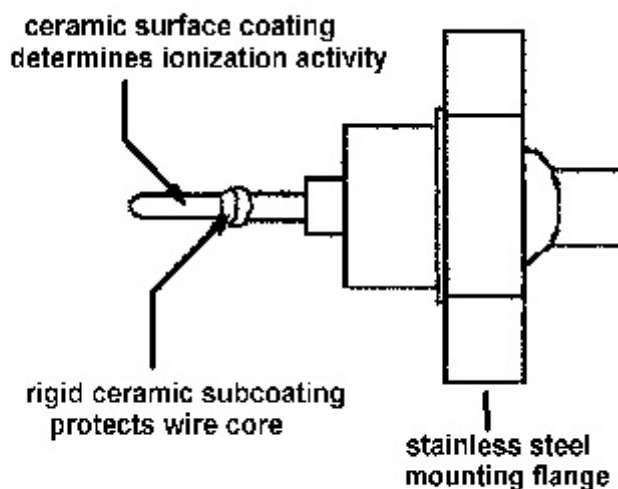
REACTOR THERMIONIC IONIZATION ANALYSIS (RTIA) - stand-alone TID or NPD transducer attached to a heated inlet reactor with a sample pump pulling ambient Air through both transducer and reactor - provides selective screening of vapors generated by Thermal Desorption and Thermal Oxidation of non-volatile constituents of liquid or solid samples - TID-1 or TID-10, TID-3, TID-7, and NPD (TID-2 or TID-4) ion sources can be used in the transducer for different selectivities.

STAND-ALONE TID, NPD, OR FID TRANSDUCERS - transducer exit connected to a sampling pump provides real time monitoring of selective or universal organic vapors in incoming ambient Air stream - selectivity determined by type of ion source element installed.

CERAMIC ION SOURCES

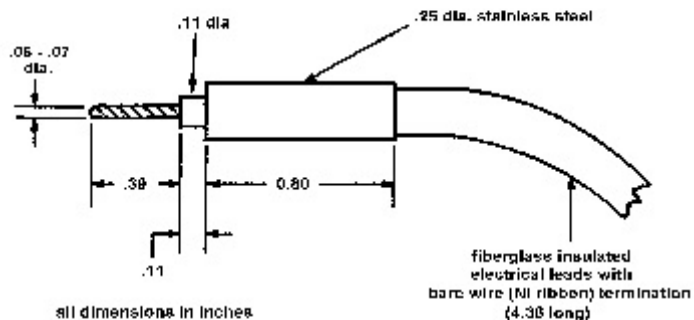
2 STANDARD MOUNTINGS

3/4 inch Hexagonal Flange Mounting



DET Ion Source Construction
(fits Agilent & DET structures)

THERMIONIC ION SOURCE 1/4 inch Tube Mounting



same ceramic coating structures and coating choices as ion sources with a hexagonal flange

(fits NPD structures designed by Thermo (Ultra) SRI, and several other GC companies.)

(Hex Flange Mounting also fits Thermo Trace 1300 NPD structure)

Optimum installation geometry for DET ion sources is a mounting attached to the end of a detector structure such that the ion source ceramic extends onto the axis of a cylindrically-shaped ion collector with sample and detector gas flows directed from the ion source tip toward the mounting.

1. Ceramic materials withstand the NPD requirement of 600 - 800° C operation without danger of softening or melting.
2. Ceramic sub-coating protects wire core from corrosive exposure.
3. Wide variety of possible surface coatings for variations in the selectivity of ionization and catalytic activity, with ongoing development of new coatings.
4. Unlimited shelf life under ambient conditions.
5. Environment friendly recycling available for used ion sources.

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HEX OR TUBE MOUNTED DET ION SOURCES

available with a Twinex connector compatible with NPD cables on Agilent 6890/7890, Thermo Trace, and Varian/Bruker GC models, and cabling from a stand-alone DET Current Supply, or with bare wire terminations (as shown)



Hex mounted ion sources with a Twinex connector are used with NPD equipment designed by Agilent for the 6890/7890 GC, and by Thermo Scientific for the Trace 1300 GC, and with all equipment designed by DET for retrofit on various GC models or for use as stand-alone transducers.

Tube mounted ion sources with bare wire terminations are used with NPD equipment designed by SRI Instruments, and are part of the ion source sub-assembly used with NPD equipment designed by Thermo Scientific for the Trace Ultra GC.

CERAMIC COATED THERMIONIC ION SOURCES by DET

Improved NPD Performance - - Extended Detection Modes

THERMIONIC IONIZATION SOURCES (BEADS) ----- \$495. each

Durable 0.062 inch diameter cylindrical ceramic structures molded about a heater wire core, and mounted on a hexagonal shaped stainless steel flange. Different ceramic additives provide a family of ion source types having different work functions for the emission of electrical charge, and different catalytic and ionizing activities. DET sources are all pre-tested for response before shipment, and are interchangeable in Agilent 6890/7890 NPD assemblies, in all DET detector assemblies, and in the Thermo 1300 NPD assembly. Standard electrical connection is a Twinex type compatible with Agilent and Thermo NPD electronics, Varian TSD electronics, as well as a stand-alone DET Current Supply. Unlike the Blos glass NPD bead, DET ceramics will not soften or melt at operating temperatures typical of NP detection. (Ion Sources are also available in a ¼ inch tube mounting, or with bare metal leads instead of the Twinex.)

TID-10 (010-910-00) - selective response to **Oxygenates** (especially **Phenols, Carboxylic Acids,** and **Glycols**), **Nitro-compounds, some Halogenates, Pyrrole functional group**, and other electronegative functionalities - operates at 400 - 600°C in inert (N₂) or oxidizing (air, O₂) gas environments – best signal-to-noise when polarized at -45 Volts or higher as available from a DET Current Supply or Thermo NPD electronics - femtogram detection for compounds like Methyl Parathion, 4-Nitrophenol, Pentachlorophenol, Heptachlor, 2,4-Dinitrotoluene, TNT when powered at high polarization – TID-10 is also the key element used in the **Catalytic Combustion Ionization (CCID)** mode which is selective to compounds containing **chains of Methylene (CH₂) groups** in Petroleum Hydrocarbons, FAMES, or Triglycerides with no response to Aromatic or Cyclo-Hydrocarbons and with discrimination between compounds containing saturate vs. unsaturated Carbon bonds. Also, **TID-9 (010-909-00)** & **TID-11 (010-911-00)** for lower & higher concentrations of TID-10 type ceramic catalyst formula. TID-10 has same response as earlier TID-1.

TID-2 (010-902-00) - NPD - selective response to **N,P compounds** - Black Ceramic coating has long life and minimal tailing of phosphorus compounds - operates at 600 - 800°C in a dilute H₂ in air gas environment - low picogram detection for NP compounds - lower cost alternative to Agilent NP sources.

TID-4 (010-904-00) - NPD - Our best coating for selective detection of **N compounds** (not recommended for P compounds because of tailing) - operates at same NPD conditions as TID-2 with 2 to 3 times better N detection than TID-2.

TID-3 (010-903-00) - Selective response to **Volatile Halogenates** - more uniform response to halogenates than TID-1 - operates at 600 - 800°C in inert (N₂) or oxidizing (air, O₂) gas environments - low picogram detection for Trihalomethanes with minimal peak tailing and greater response for Br versus Cl – best sensitivity when polarized at -45 Volt or higher.

TID-5 (010-905-00) - Halogen selective detection - more uniform response than TID-3 - operates at same temperatures and gas environment conditions as TID-2, but with a polarization voltage of -45 V or higher – also provides exceptional **selectivity for Br and I versus Cl** when configured with a stoichiometric mixture of Hydrogen and Air (e.g., H₂ = 5, Air = 12.5, N₂ makeup = 30 mL/min).

TID-6 (010-906-00) - P selective detection with suppressed N response - uses much higher Hydrogen, Air, and Nitrogen flows than the TID-2 and TID-4 NPD modes, and uses a detector structure with an internal upstream restrictor to prevent flashback to a self sustaining flame (e.g., H₂ = 20, Air = 200, N₂ makeup = 100 mL/min).

TID-7 (010-907-00) - Green Ceramic for **Halogenated Pesticides, PCBs** - operates in N₂, Air, or O₂ - best sensitivity when polarized at -45 V or higher.

CFID (020-901-00) – High work function for operation downstream of a flame in a **Remote FID** detection mode which is selective to **compounds containing P, Pb, Sn, or Si atoms**. Selectivity improved by using a Hydrogen-Methane fueled flame.

FID Probe (020-902-00) – Uncoated Pt alloy wire used for **Universal Detection** in **FID** or **HWCID** (Hot Wire Combustion Ionization) mode.

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DET RETROFIT NPD/TID/CCID/FID HARDWARE FOR DIFFERENT GC MODELS

DET retrofit hardware is easily mounted onto an existing NPD or FID base, and uses the existing heater and pneumatics controls connected to that base. All DET retrofits consist of a tower structure featuring a common concentric cylinder geometry that positions an electrically-heated ion source in the center of a collector electrode, with top access for easy interchange of ion sources. Mounting on the bottom of the tower is custom designed to be compatible with the detector base on the GC being retrofitted. DET hardware is compatible with all DET ion source elements so that NPD, TID, CCID, and FID modes of detection can be accommodated with the same equipment.

THERMO TRACE ULTRA GC (part 010-860-55, price \$2340). Replaces the Thermo NPD hardware which has a side mounted ion source, with a more optimum concentric cylinder geometry and a top mounted ion source. Hardware includes a ceramic tipped jet. Any DET ion source priced at **\$495** each can be accommodated. Hardware is fully compatible with Thermo NPD electronics, and the combination provides the most versatile NPD, TID, CCID, FID equipment currently available. Tandem TID hardware is also available for 2 simultaneous signals from one sample, as well as Remote FID hardware for added selectivity of P, Pb, Sn, Si compounds.

VARIAN/BRUKER/SCION GC MODELS (part 010-860-20, price \$2100). Replaces the Varian/Bruker/Scion TSD/NPD hardware which has a side mounted NP bead, with a more optimum concentric cylinder geometry and a top mounted ion source. Hardware includes a ceramic tipped jet that seals into the detector base with a standard stainless steel ferrule rather than the crushable Vespel/Graphite ferrule required by a Varian/Bruker/Scion jet. Varian retrofits can use any standard DET ion source priced at **\$495**, and these can be powered with Varian TSD electronics. Bruker/Scion retrofits require custom low resistance ion sources priced at **\$700** in order to be powered by Bruker/Scion TSD/NPD electronics. Signal-to-noise for TID, CCID, FID modes can be substantially improved by powering ion sources with a stand-alone DET Current Supply described below. For Bruker/Scion GCs, powering ion sources with the DET Current Supply requires use of a simulated bead (**\$175**) to avoid Bruker/Scion "bead open circuit" sensing, although the DET supply then allows DET's lower price (**\$495**) standard ion sources to be used. Tandem TID and Remote FID hardware assemblies are also available.

SRI INSTRUMENTS GC MODELS (part 050-864-98, price \$2340). Replaces the SRI NPD/FID hardware with a more optimum concentric cylinder geometry and an end mounted ion source. Hardware includes a ceramic tipped jet. Ion sources used with this hardware have bare wire terminations (**priced at \$460**), and any type DET ion source can be accommodated. SRI's existing NPD or FID electronics can be used to power the ion sources and measure signals.

AGILENT 6890/7890 NPD MODELS

All DET ion sources are compatible with mounting into existing Agilent 6890/7890 NPD hardware. Agilent electronics suffice for NPD operation, but substantial improvement for TID and CCID modes of detection is achieved by substituting a stand-alone DET Current Supply to power ion sources with a higher polarization voltage. DET also recommends replacing the small orifice Agilent NPD jet with a wide bore jet as described below.

DETECTOR CURRENT SUPPLY (part 001-901-01, 115Vac) - - - - - \$2244. each

Stand-alone module provides heating current and a selection of -5, -15, or -45 V polarization voltages for DET thermionic sources or the Agilent NP source. Recommended for use in place of the Bead Voltage supply on the Agilent NPD because it provides more stable Constant Current heating power for thermionic sources versus the Constant Voltage power provided by the Bead Voltage supply. Also, for modes of detection other than NP, the higher polarizations available from the DET supply provide as much as a factor of 10 improvement in signal-to-noise versus the fixed low polarization available from the Agilent supply. The DET supply also includes a green/red status light to immediately indicate that the source has burned out or the source power cable is not properly connected.

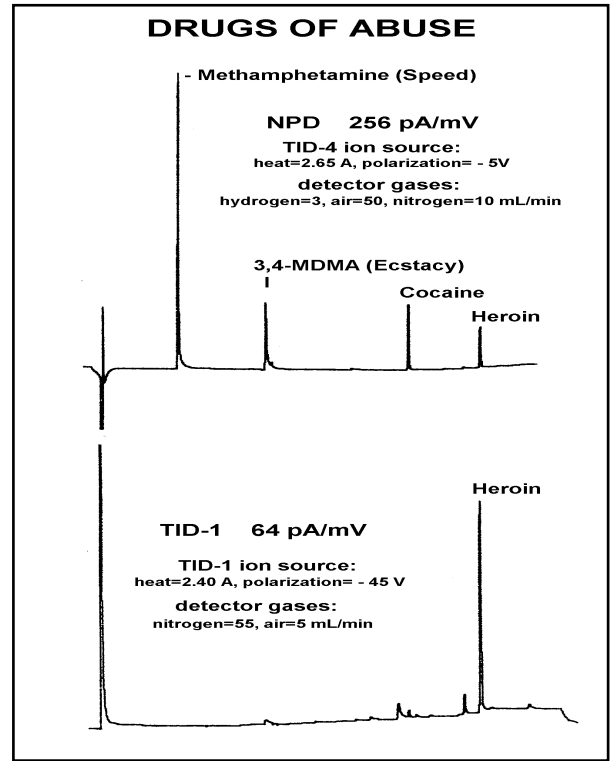
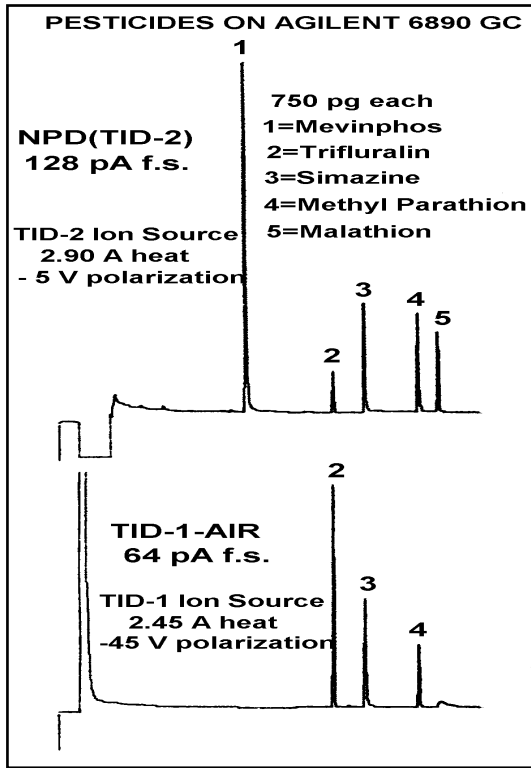
WIDE BORE JET & COLUMN SPACING KIT

Allows capillary columns of 0.53 mm diameter or less to be inserted through the jet to a termination close to the ion source as defined by a spacer tool. Eliminates sample degradation from interaction with jet metal; eliminates jet clogging from sample matrices; and eliminates the need to ever replace the jet.

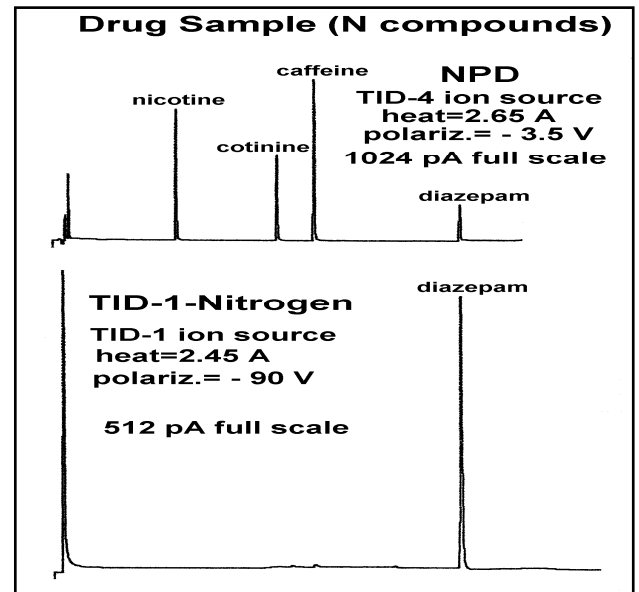
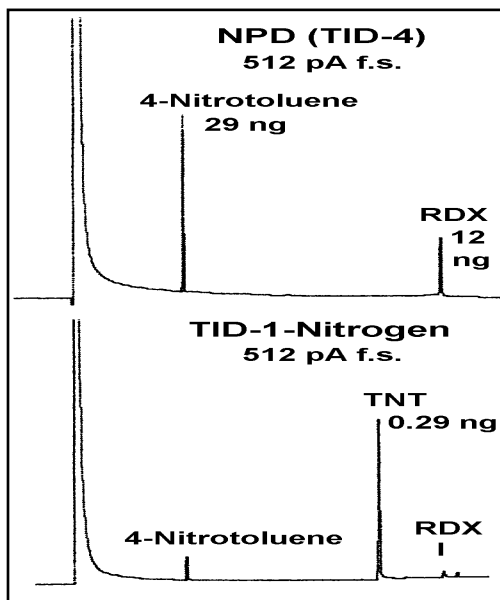
(010-886-13) - column spacer & 64 mm long jet for Agilent's adaptable fitting NPD base - **\$ 190. each**

(010-887-13) - column spacer & 43 mm long jet for Agilent's dedicated capillary base - **\$ 250. each**

THERMIONIC SURFACE IONIZATION DETECTION FOR GC selectivity for NITROGEN and PHOSPHORUS compounds (NPD)



TID-2 Black Ceramic designed for sharp P peaks TID-4 White Ceramic designed for best N response
TID-1 (TID-10) mode is more compound selective than NPD



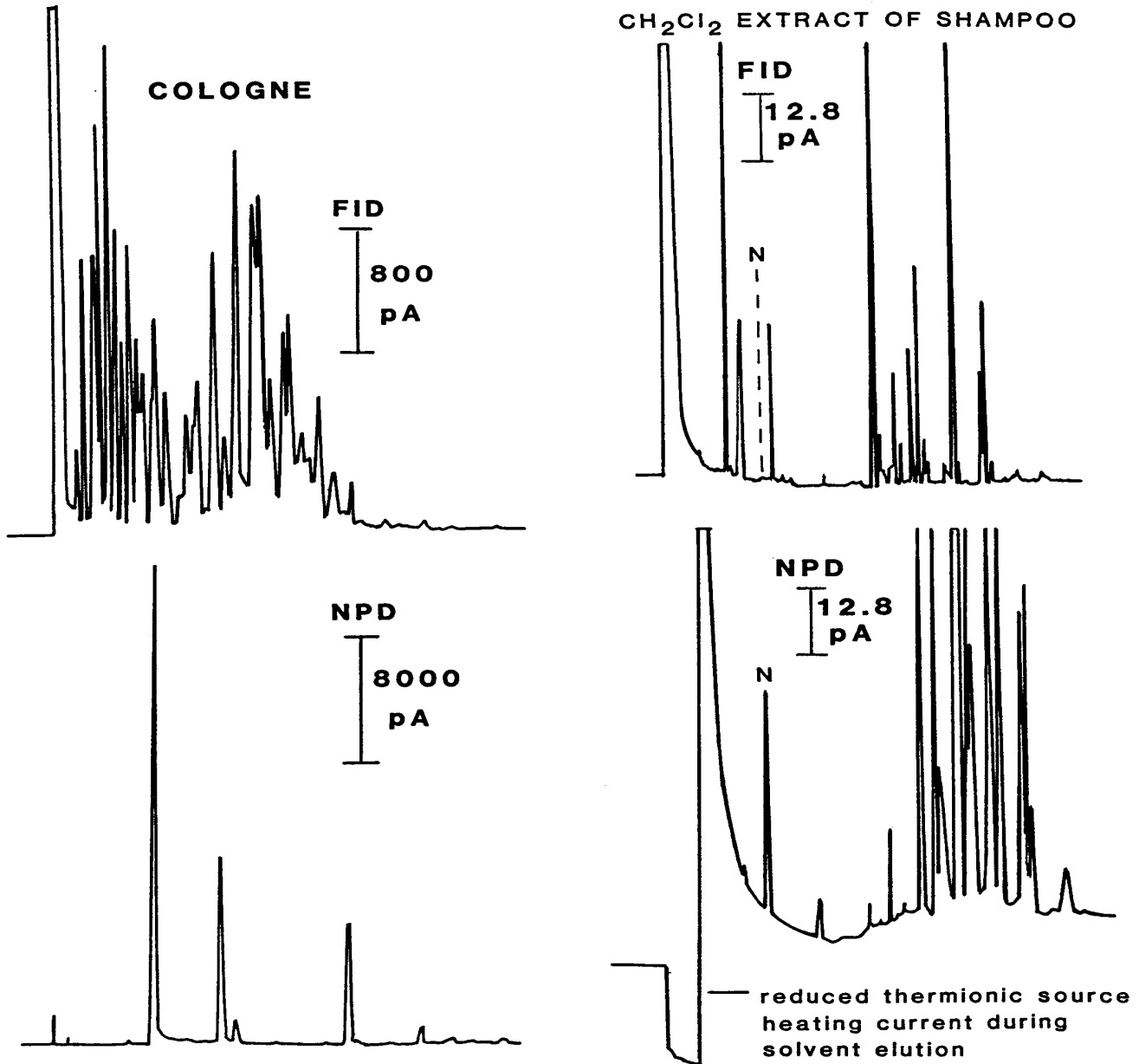
Explosives detection – NPD detects all N compounds – TID-1 mode has exceptional sensitivity for TNT

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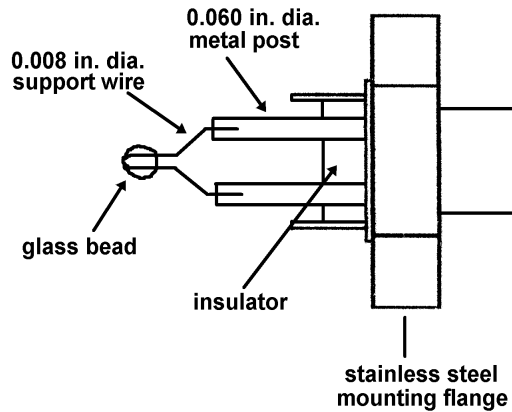
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NITROGEN-PHOSPHORUS SELECTIVE DETECTION from DET

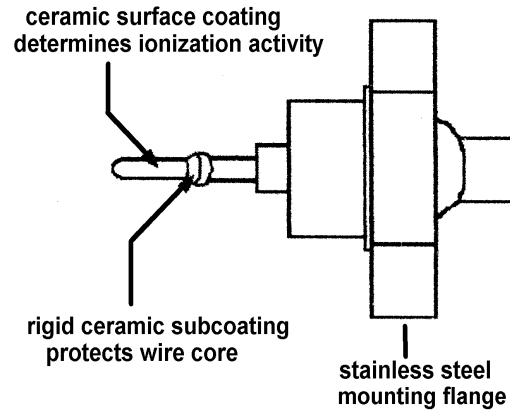


Compared to FID analyses of complex samples, an NPD provides both selectivity and detectivity advantages for trace N and P constituents. Selectivity allows shorter analysis times because undetected sample matrix components do not need to be well resolved chromatographically.

COMPARISON OF ION SOURCES FOR AGILENT NPD

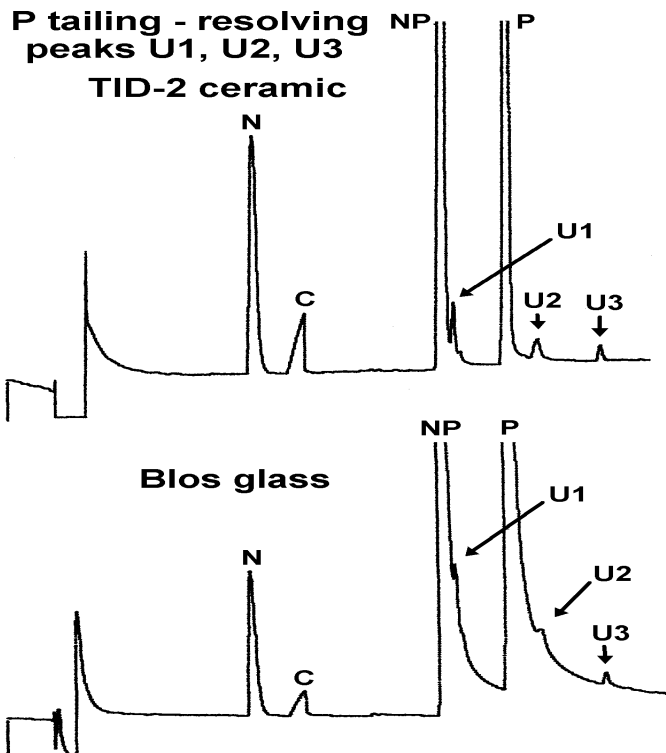


BloS Bead for Agilent NPD



**DET Ion Source Construction
(fits Agilent & DET structures)**

BloS NP ion source is a small Glass Bead deposited on the end of a loop of bare support wire. DET ion source is a more rigid cylindrical structure comprised of layers of ceramic materials coated over an inner wire core. NP detection requires the ion source to be heated to temperatures in the range of 600 - 800° C in order to ignite the Hydrogen-Air chemistry. At those temperatures, the BloS Glass is in a softened physical state, whereas DET Ceramics remain solid. In operation, a DET NP ion source has an electrical resistance of 1.2 Ohms versus 0.2 Ohms for the BloS Bead. That BloS resistance is not much greater than the resistance of the attached external wiring leads and connectors, so those components get hotter than is the case with DET ion sources.



PHOSPHORUS PEAK TAILING

Tailing of Phosphorus peaks has been a notorious characteristic of many past NP ion sources. DET's Black Ceramic coating is formulated to eliminate P tailing. This allows detection of small peaks eluting right after large peaks, and allows rapid recovery to baseline so that another analysis can be run soon thereafter.

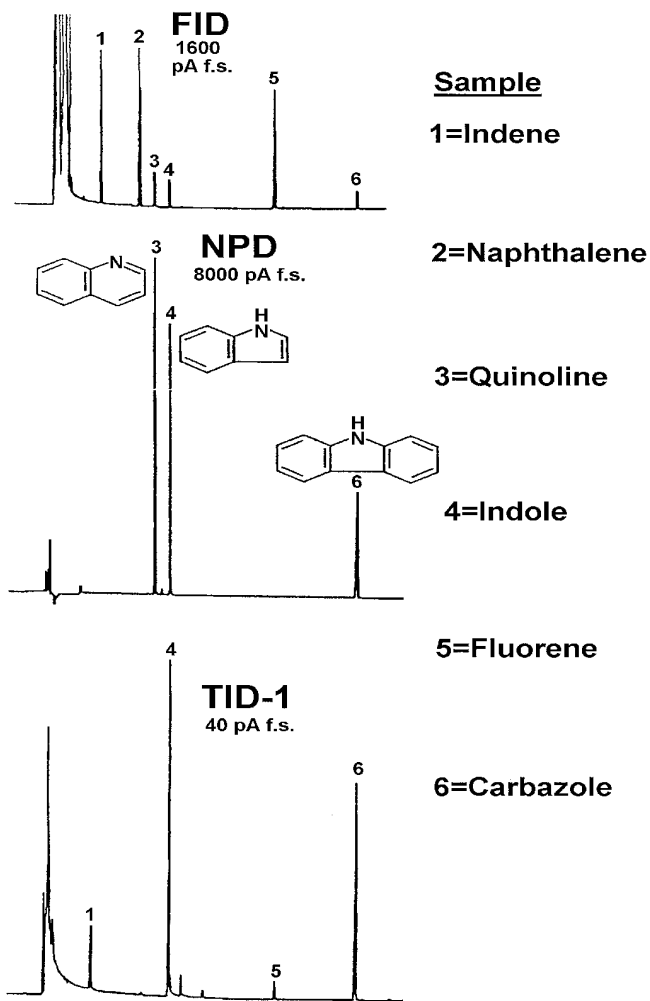
All DET ion sources are conditioned and tested prior to shipment, so they are ready for use when installed in an instrument. This means minimum instrument downtime when replacing an ion source. DET ceramics also have unlimited shelf life when stored under ambient conditions.

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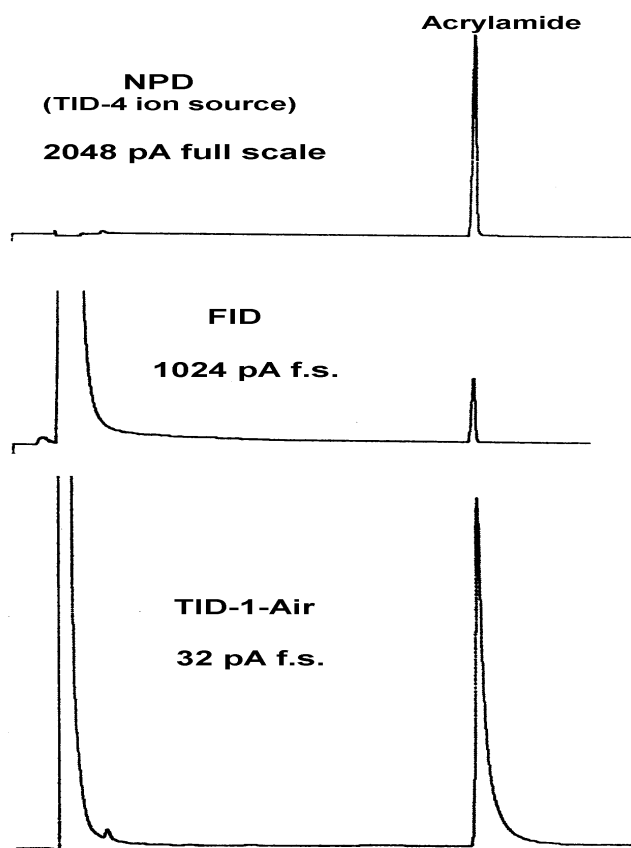
DETector Engineering & Technology, inc.
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Nitrogen Heterocycles Pyridine-Pyrrole Functional Groups



NPD Detection of Acrylamide

50 ng Acrylamide in Methanol Solvent



Compared to an FID, NPD provides both a selectivity and sensitivity improvement. While NPD detects all the N components, more selective TID-1 mode (TID-10 is same) detects just the Pyrrole components, Indole and Carbazole, due to selectivity for the 5 member N ring. Note that TID-1 also selectively detects at a lower response the 5 member C ring compounds, Indene and Fluorene.

Another example where an NPD provides improvement in both selectivity and sensitivity versus an FID for the detection of N compounds. Of the 3 different modes of detection shown for the analysis of this compound, NPD is clearly the preferred mode.

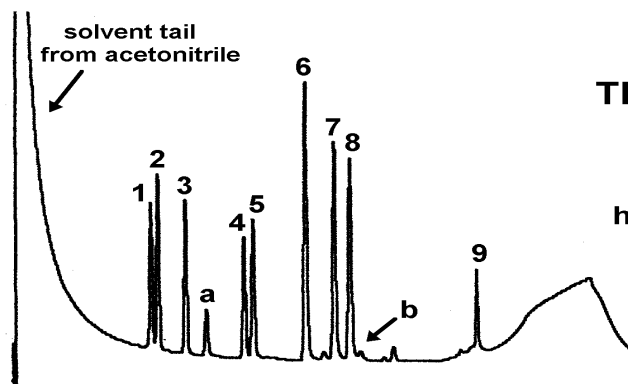
Thermo Trace Ultra GC - DET NPD/TID Hardware - Thermo NPD Electronics

**EXPLOSIVES
SAMPLE**
(N compounds in an
N solvent)

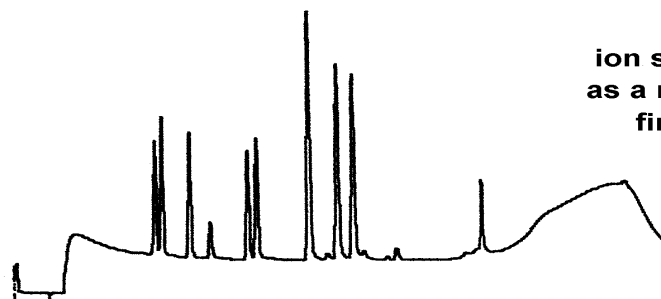
Solvent:
1% acetonitrile in
methanol

10 ng each

1=1,3-dinitrobenzene
2=2,6-dinitrotoluene
3=2,4-dinitrotoluene
4=1,3,5-trinitrobenzene
5=TNT
6=RDX
7=4-amino-2,6-dinitrotoluene
8=2-amino-4,6-dinitrotoluene
9=HMX
a,b - unidentified

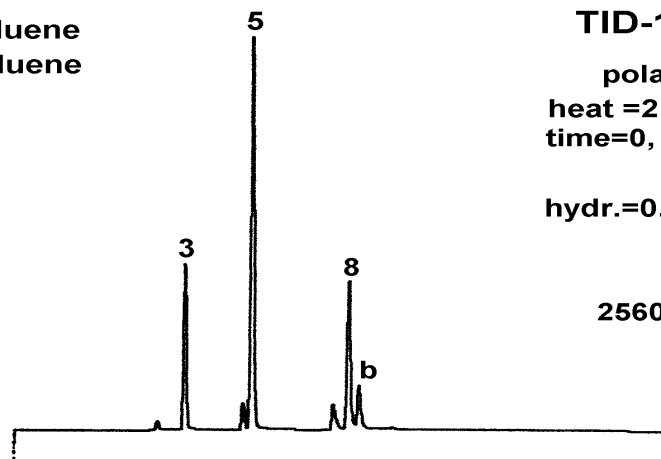


NPD
TID-4 Ion Source
heat=2.65 A
polarization=3.5 V
hydr.=3, air=50, nitr.=5
256 pA full scale



NPD
ion source heat turned off
as a run table event during
first 1.0 minute

256 pA f.s.

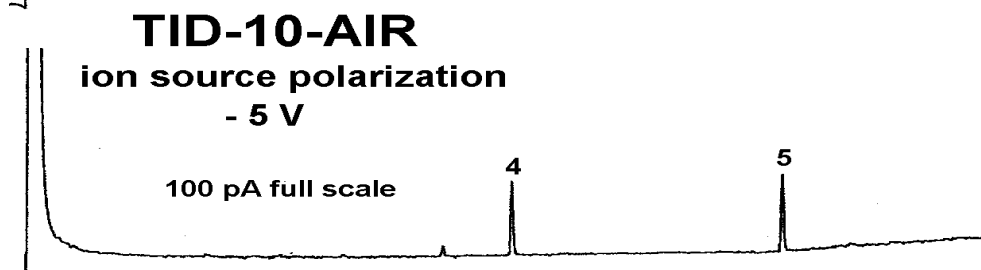
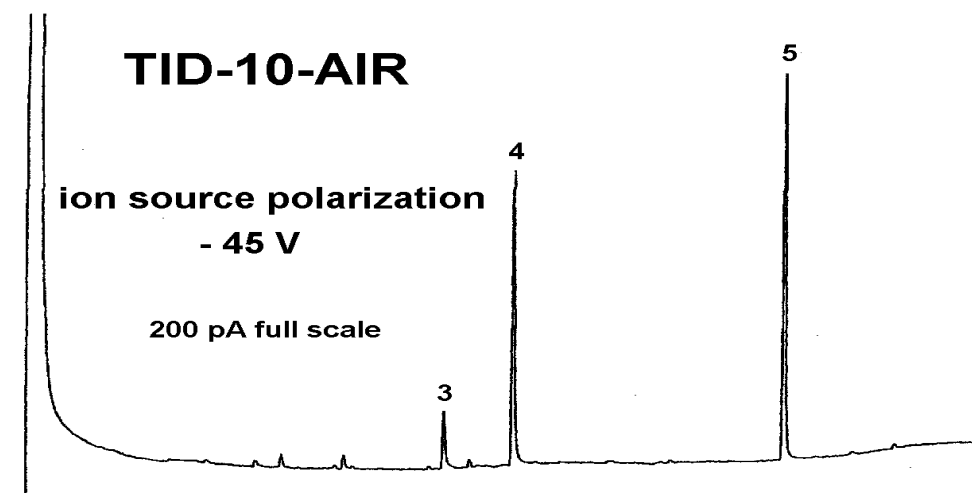
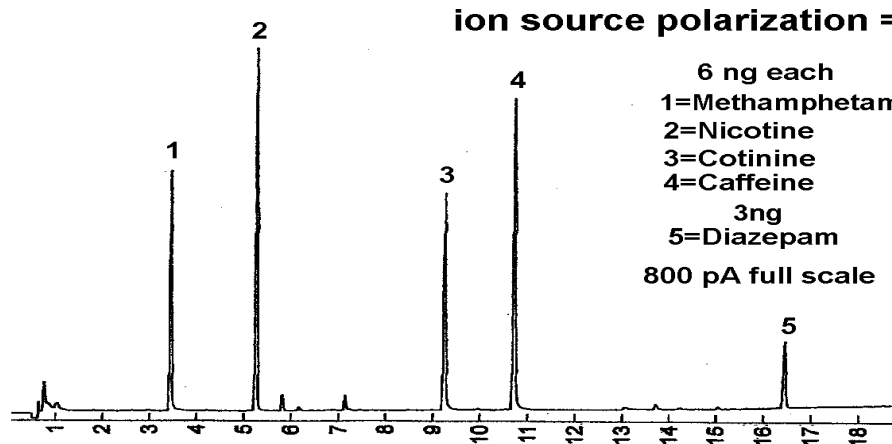


TID-10 Ion Source
polarization=99 V
heat =2.35 A turned off at
time=0, on at time=1.0 min.
hydr.=0.5, air=50, nitr.=10

2560 pA full scale

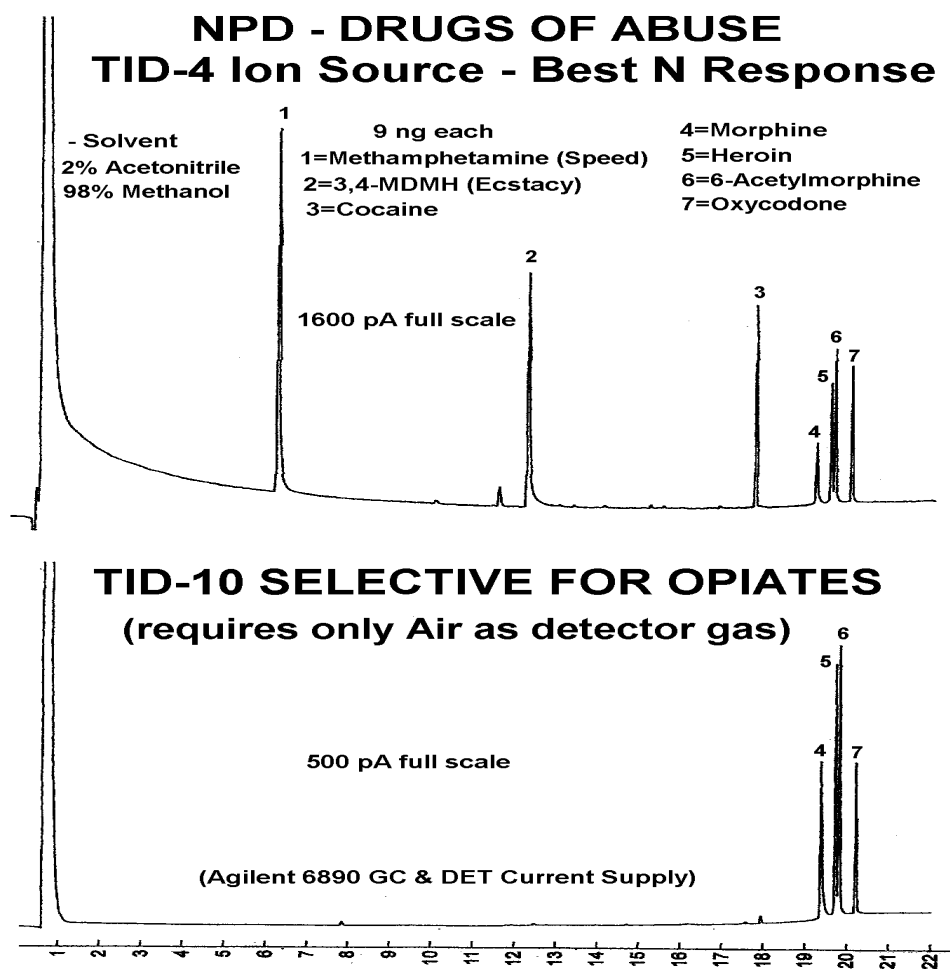
Comparison of NPD vs. TID-10 modes of selective detection - same hardware structure and electronics with different type ion sources and different type detector gases. NPD detected all the explosive compounds, while TID-10 provided high selectivity and very large responses to compounds 3, 5, and 8 which contained a Nitro (NO₂) group located in a para position relative to other functionalities in the compound's molecular structure. This was an analysis of trace N compounds in an N compound solvent. Thermo's NPD electronics allowed ion source heating current to be turned off during solvent elution to eliminate the large solvent peak and peak tail.

NPD (TID-4 Ion Source) - DRUG SAMPLE



(Agilent 6890 GC & DET Current Supply)

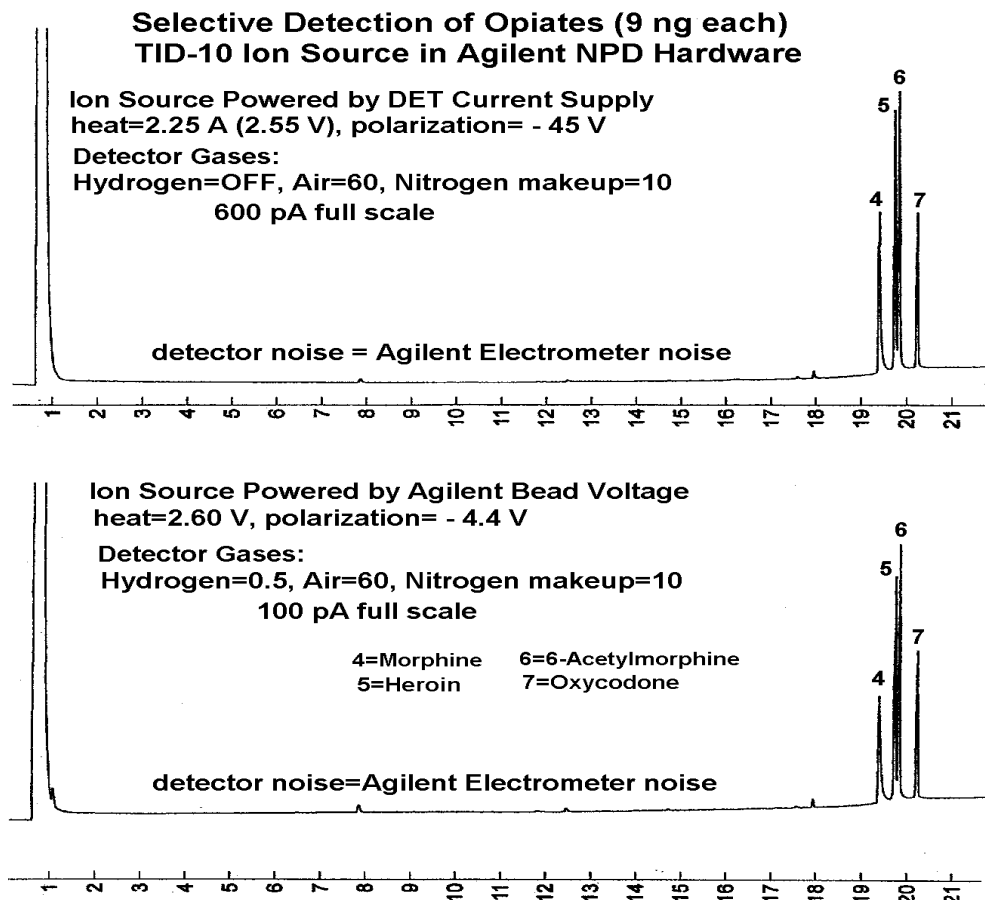
In addition to Opiates, TID-10 detection also responds to certain other types of drug compounds. For this sample, Diazepam and Caffeine both responded well with somewhat smaller response to Cotinine. The data also demonstrate again that a low ion source polarization that is typical of optimum NP detectors, produced lower signal magnitudes, although selectivity was still retained.



The NPD provided signals of larger absolute magnitudes, but background and noise were also about a factor of 3 higher, so TID-10 detectivity (signal-to-noise) was close to that of the NPD.

A stand-alone DET Current Supply was used instead of the Agilent Bead Voltage power for the TID-10 ion source to obtain larger signals with a higher polarization between the ion source and collector electrode in the detector.

Two of the standards used in preparing this drug mix came in an Acetonitrile solution which caused the solvent peak tailing displayed in the NPD data. Conversely, TID-10 is responsive to Methanol, but the Air detector environment resulted in rapid recovery from the effect of that solvent component. For NPD applications involving detection of trace N compounds in a solvent also comprised of an N compound, a useful practice is to turn off the Hydrogen-Air chemistry during solvent elution by reducing either the ion source heat or Hydrogen flow as a GC time event. This usually greatly reduces long term tailing from the solvent.



TID-10 detection signals get bigger with increasing magnitude of the polarization voltage between the ion source and collector electrode. These data show the difference between Opiate signal magnitudes when a DET Current Supply at - 45 V polarization was used for ion source power vs. using Agilent's Bead Voltage which is fixed for optimum NP detection at a polarization of - 4.4 V. Background signals for both sets of data were low so that detector noise was the noise of the Agilent Electrometer. With the DET supply, signal magnitudes and detectivity were about 6 times better than provided with the Agilent supply. Of the various GC manufacturers that use DET ion sources in their NPDs, only Thermo has NPD electronics that can provide polarization voltages in the range of 0 to - 100 V in order to achieve optimum performance in all possible modes of selective detection.

TID-10 detection does not require Hydrogen in the detector gases. However, when using the Agilent Bead Voltage, some gas flow is required through the gas lined labeled "Hydrogen" in order to get a GC "Ready" state. For the data above, a minimal Hydrogen flow of 0.5 mL/min was used. It would have been better to supply Nitrogen through both "Hydrogen" and Makeup lines, but we wanted to retain flexibility for quickly changing between TID-10 and NPD modes.

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CATALYTICALLY ACTIVE TID-10 CERAMIC ION SOURCE



Converts Agilent 6890/7890 NPD and Thermo Trace 1300 NPD equipment to **Oxygenate selective detection**. Requires only Nitrogen or Air as the detector gas.

O is the most common occurring heteroatom in organic chemistry. Addition of O selectivity to NPD equipment greatly expands the potential application market for that equipment. (Example applications include **foods, flavors, fragrances, beverages**.) Within the O selectivity of TID-10 ion sources is added response enhancement for certain Oxygenates relative to others. **Carboxylic Acids, Phenols, and Glycols** have especially large responses relative to Alcohols, Ketones, etc.

In addition to O selectivity, a TID-10 ion source is the key element used in DET's exclusive **Catalytic Combustion Ionization Detection** mode (CCID). CCID provides selectivity to high concentrations of compounds containing chains of Methylene (CH_2) functional groups (examples, **Alkanes, Alkenes, FAMES, Triglycerides**). CCID requires a detector gas environment containing Oxygen (i.e., Air, O_2) and involves the sample compound itself fueling a momentary burst of flame ionization as the compound impacts the heated TID-10 ion source. CCID **does not respond to Aromatic or Cyclo-hydrocarbon compounds**, so great simplification is achieved for detecting selected constituents in complex Petroleum and Biofuel chromatograms. Through judicious adjustments of Oxygen concentration in the detector gases, additional **differentiation is achieved between Linear vs. Branched compounds, Saturates vs. Unsaturation, and Mono-Unsaturation vs. Poly-Unsaturation**.

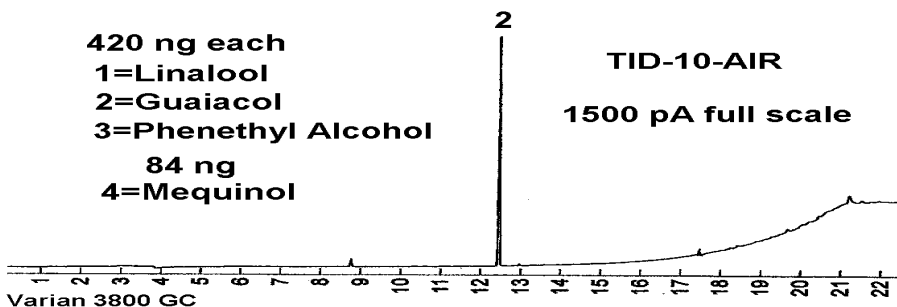
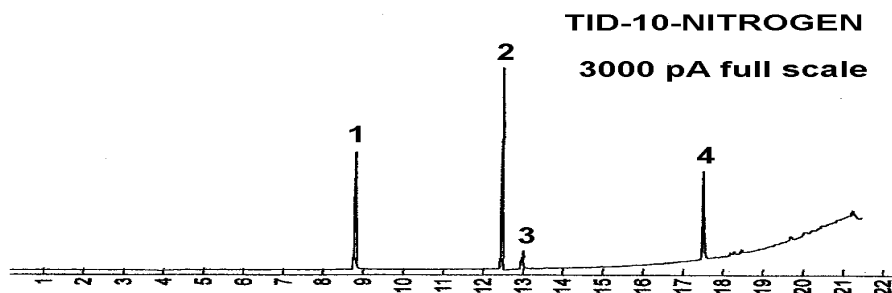
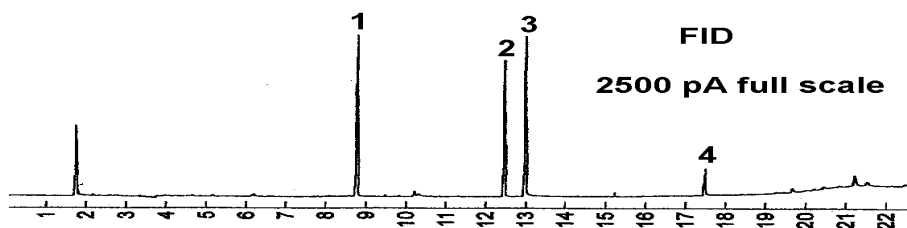
TID-10 Ion Source, part 010-910-00, price \$495 USD

DET

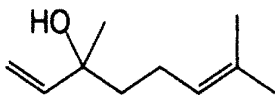
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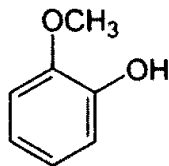
TID-10 DETECTION OF OXYGENATES



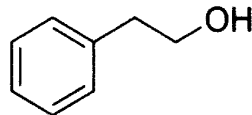
Linalool



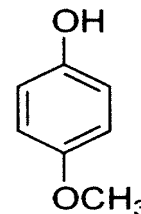
Guaiacol



Phenethyl Alcohol



Mequinol



These data demonstrate that TID-10 Oxygenate responses often depend on the molecular structure of the sample compound. Also, the simple change from an inert Nitrogen detector gas environment to an oxidizing Air environment can further change the selectivity of TID-10 response.

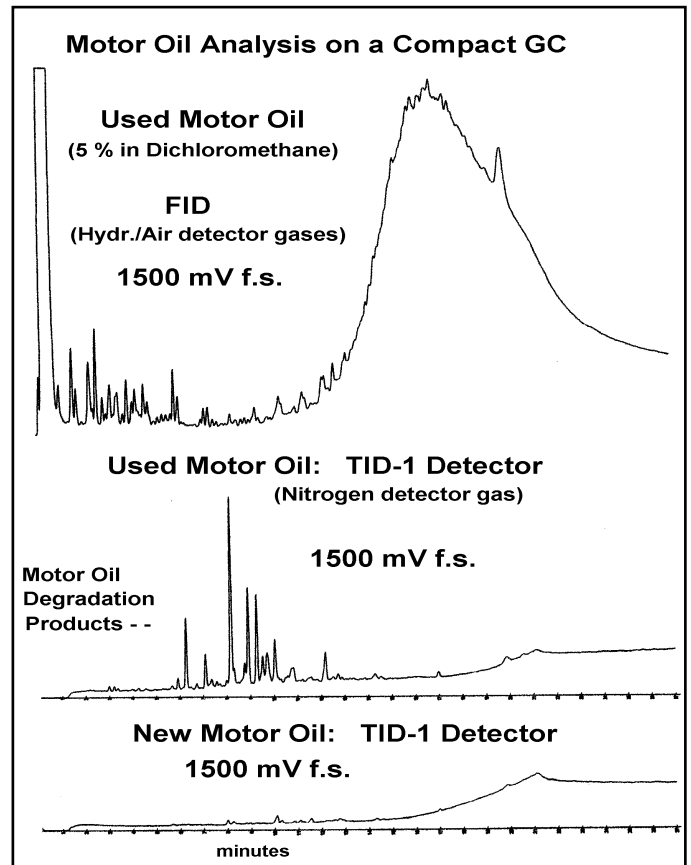
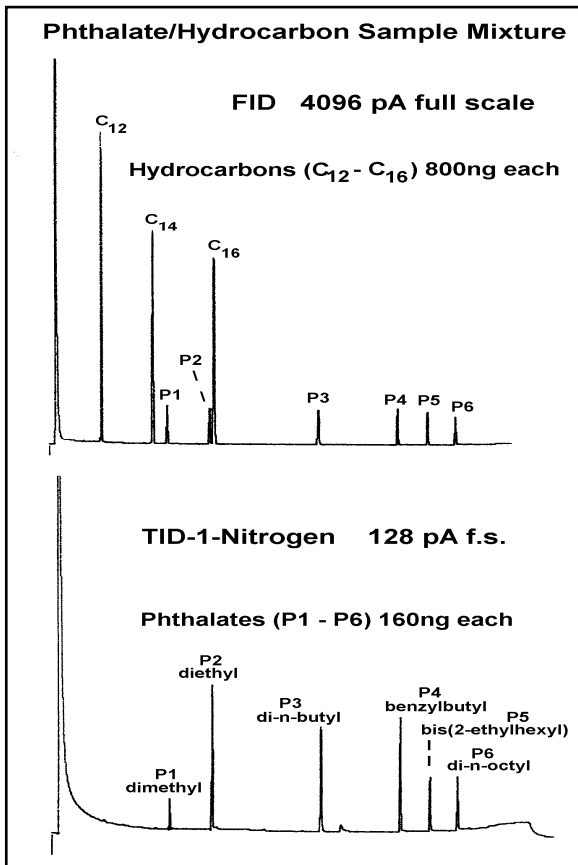
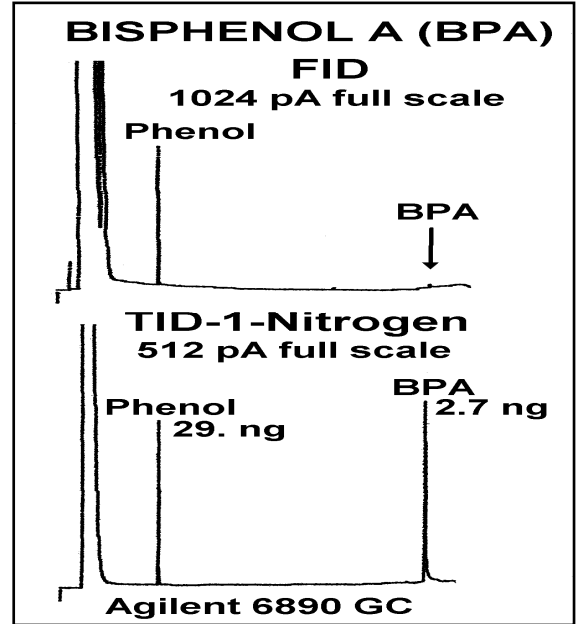
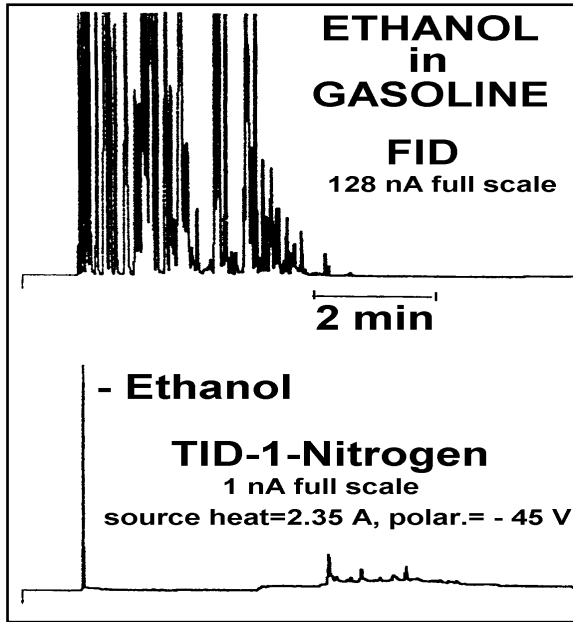
In comparison to the FID data, TID-10-Nitrogen responded to the Oxygenated components with a different selectivity, as well as with larger signal magnitudes.

DET

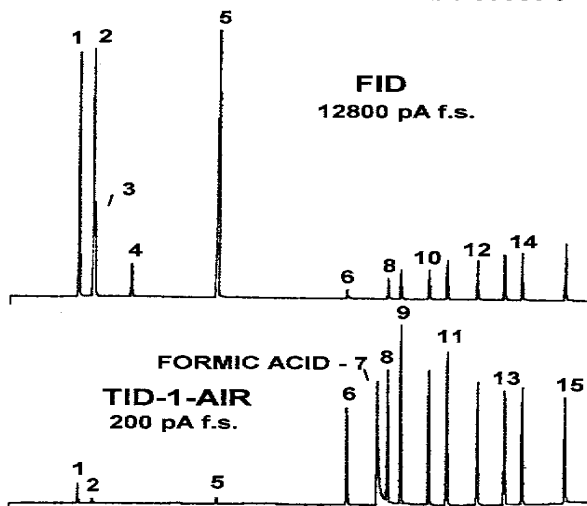
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THERMIONIC SURFACE IONIZATION DETECTION FOR GC selectivity for OXYGENATED compounds (Alcohols, Phenols, Carboxylic Acids, Glycols, Phthalates, Water, etc.)



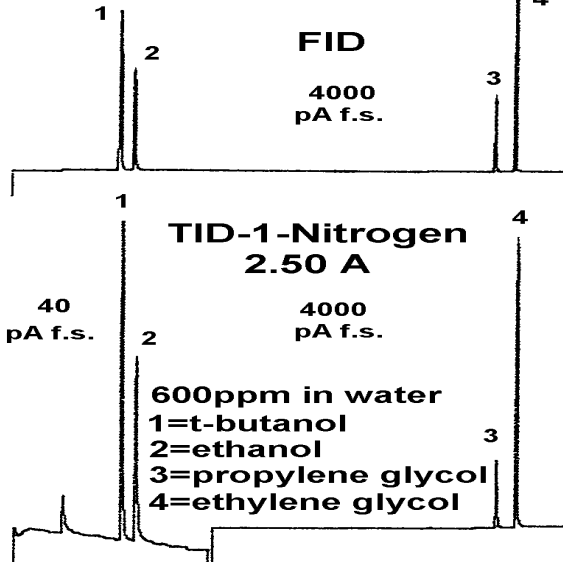
CARBOXYLIC ACID SELECTIVITY



Water solution: 1=Ethanol (1%), 2=Ethyl Acetate (1%), 3=Benzene (0.15%), 4=Toluene (0.1%), 5=iso-Pentanol (1%), 6=Acetic Acid (0.1%), 7=Formic Acid (0.1%), 8=Propionic Acid (0.1%), 9=iso-Butyric Acid (0.1%), 10=Butyric Acid (0.1%), 11=iso-Valeric Acid (0.1%), 12=n-Valeric Acid (0.1%), 13=iso-Caproic Acid (0.1%), 14=n-Caprioc Acid (0.1%), 15=Heptanoic Acid (0.1%).

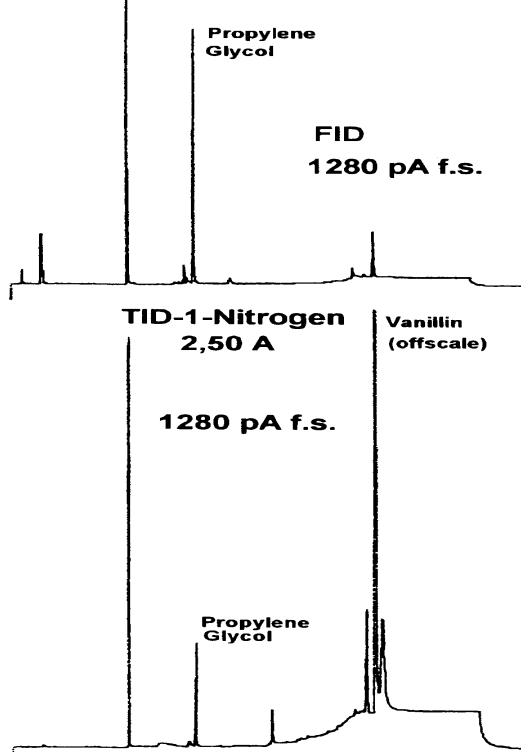
Large TID-1 Carboxylic Acid response vs. Alcohols, including response to Formic Acid which is not detected by an FID. Detector gas environment of Air further suppresses Alcohols vs. Acids responses.

ALCOHOLS/GLYCOLS

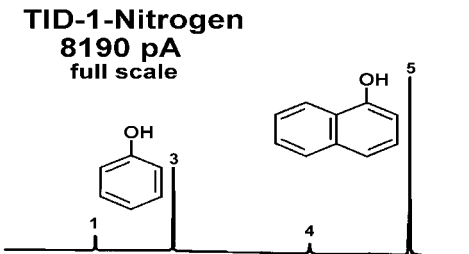
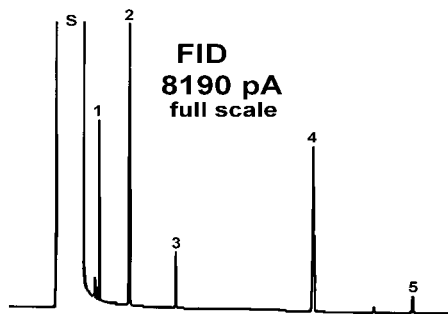


Large TID-1 response to Glycols vs. Alcohols. TID-1 Glycol response comparable to or greater than FID response.

IMITATION VANILLA



TID-1 is extremely sensitive to Vanillin. Much larger response than FID.



S=benzene, 1=cyclopentanol
 2=p-xylene, 3=phenol,
 4=n-decanol, 5=1-naphthol

Large TID-1 response to Phenols vs. Alcohols. TID-1 Phenol response comparable to or greater than FID response. Polycyclic 1-Naphthol response bigger than Phenol response.

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

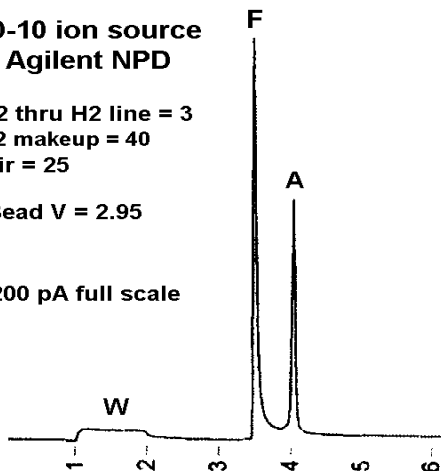
FORMIC (F) & ACETIC (A) ACIDS 0.1 % each in Water (W)

TID-10 ion source
in Agilent NPD

N₂ thru H₂ line = 3
N₂ makeup = 40
Air = 25

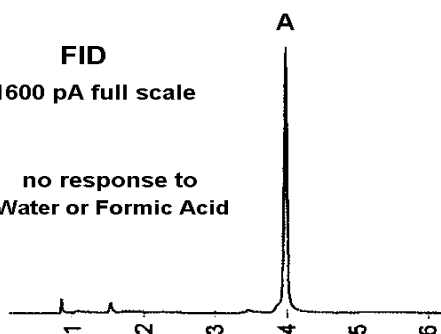
Bead V = 2.95

1200 pA full scale



FID
1600 pA full scale

no response to
Water or Formic Acid

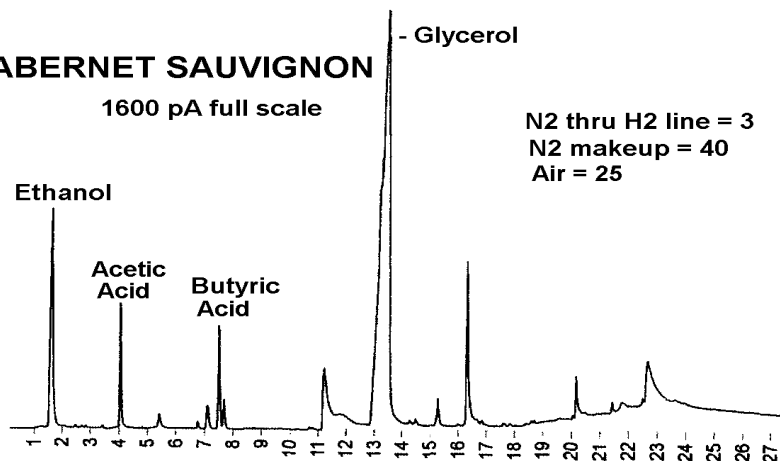


TID-10 Ion Source in Agilent NPD - Wines 0.17" ID collector insert, Bead V = 3.04

CABERNET SAUVIGNON

1600 pA full scale

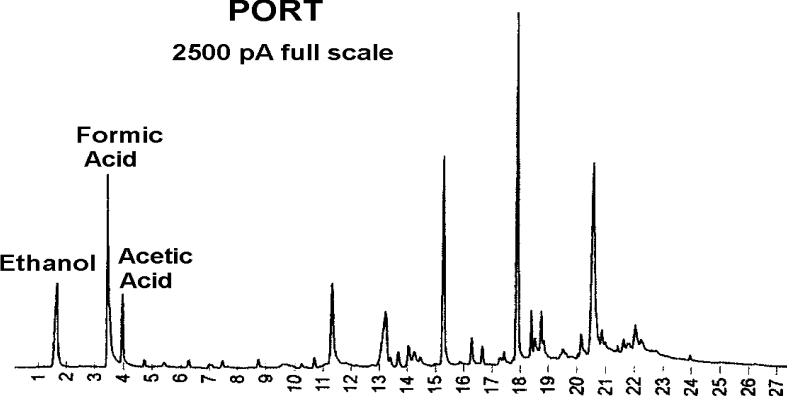
N₂ thru H₂ line = 3
N₂ makeup = 40
Air = 25



PORT

2500 pA full scale

Formic Acid
Ethanol
Acetic Acid



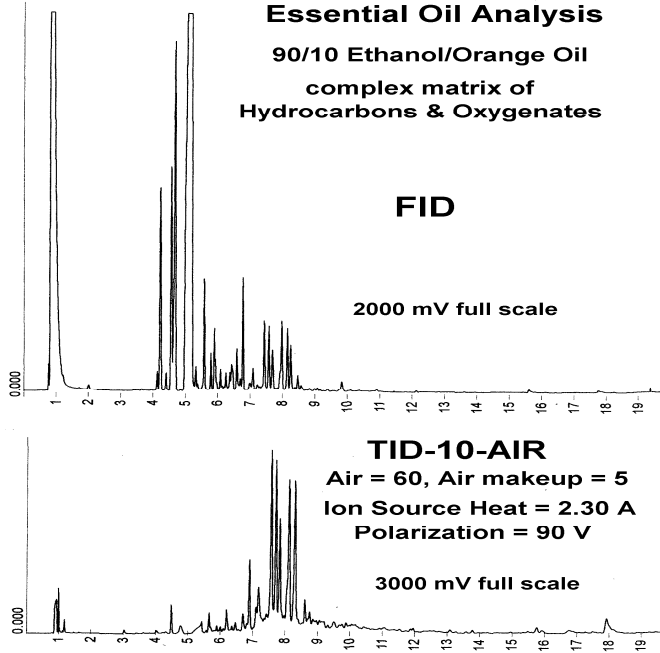
Selective detection of Oxygenates by means of the TID-10 Thermionic Surface Ionization process produces different magnitudes of response depending on the Oxygenate class. Volatile Acids, Phenols, and Glycols have especially large responses relative to other Oxygenates such as Alcohols. Vanillin and Methyl Salicylate are other examples of 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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THERMIONIC SURFACE IONIZATION – SELECTIVE DETECTION OF OXYGENATES

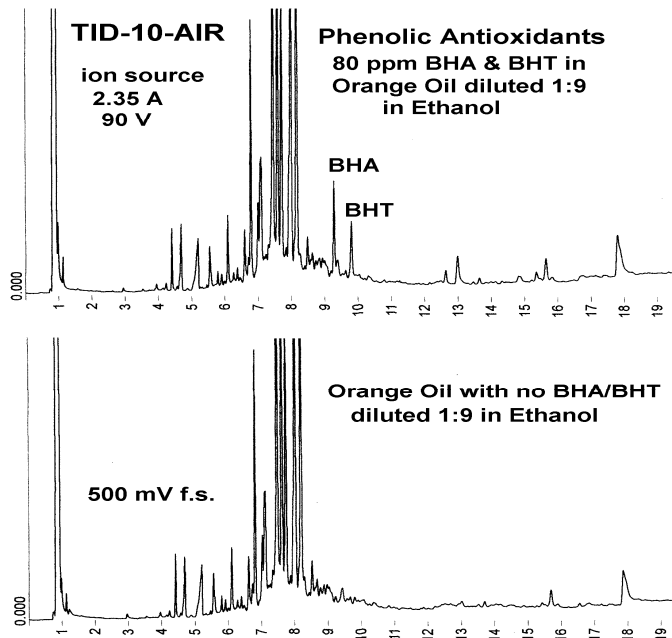


ESSENTIAL OILS

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 "H₂", 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.

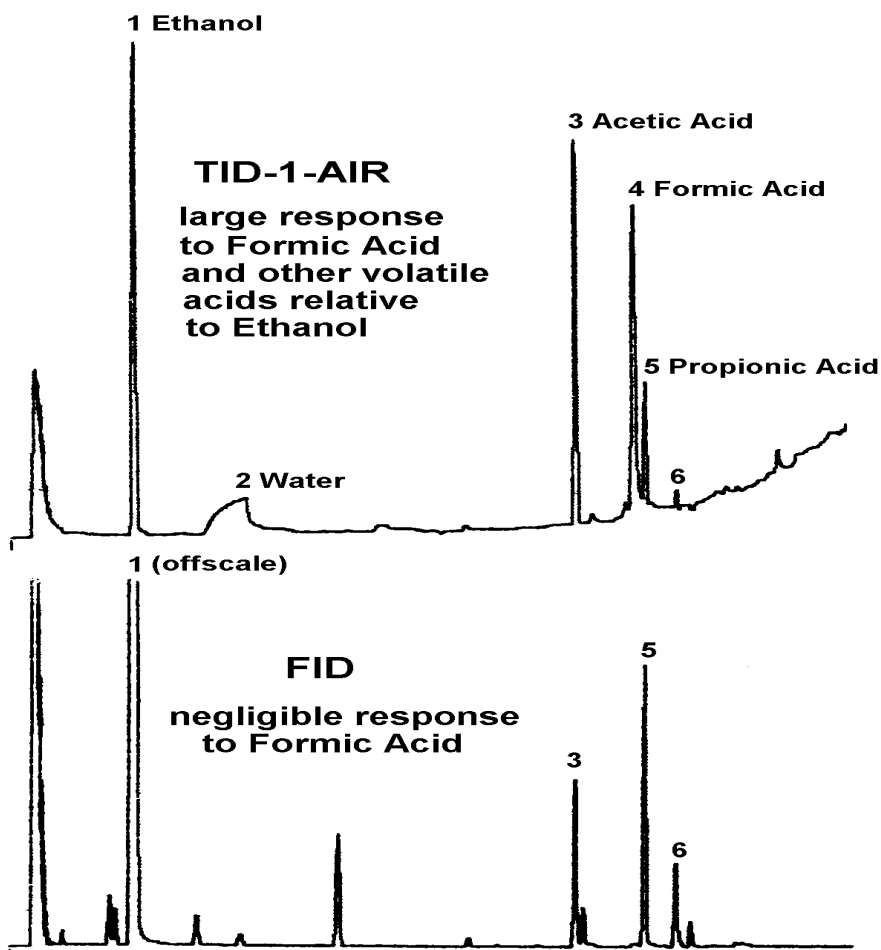
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TID-1 (TID-10) SELECTIVITY FOR VOLATILE CARBOXYLIC ACIDS IN WINE (TID-10 is a recent, more robust version of a TID-1 Ceramic Ion Source)

FORMIC ACID IN WINE (Johannesburg Reisling)

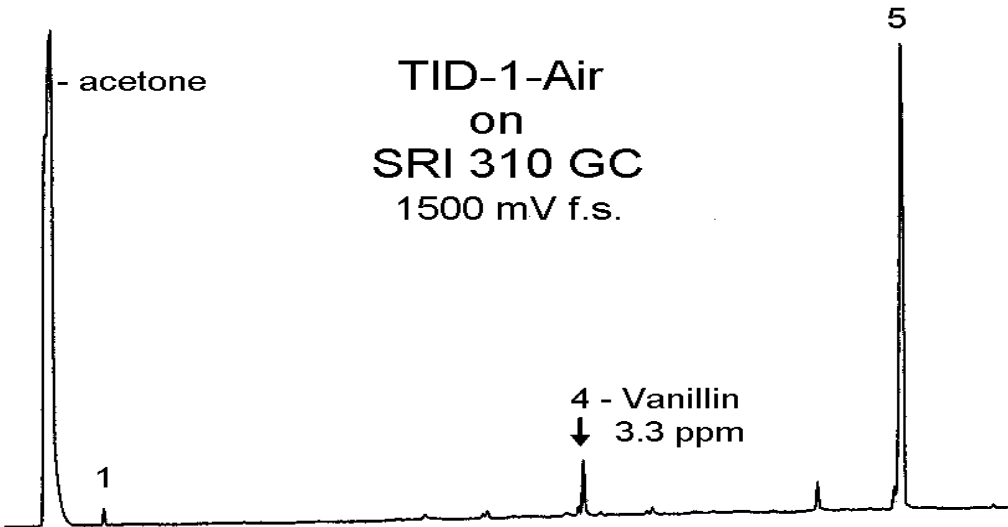
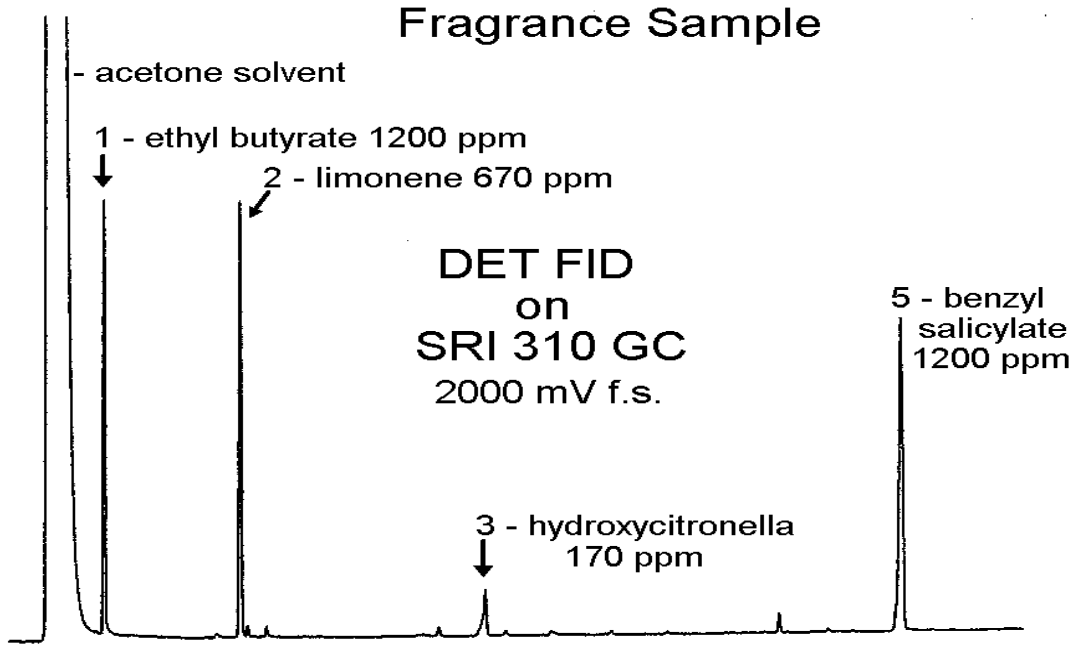


TID-1 (TID-10) ion sources operate in either an inert Nitrogen or oxidizing Air detector gas environment, and provide selective detection for Oxygenated compounds with especially large signals for Carboxylic Acids, Phenols, and Glycols versus other classes of Oxygenates. For analyses of alcoholic beverages, an Air environment provides some suppression of the large Ethanol peak relative to the Volatile Carboxylic Acids. Response includes Formic Acid and Water which have negligible responses with an FID. Glycerol and Vanillin constituents in Wine are also detected with enhanced sensitivity. Also, using an Air environment to suppress the Ethanol peak allows detection of a trace peak of Diacetyl which elutes on the tail of the Ethanol.

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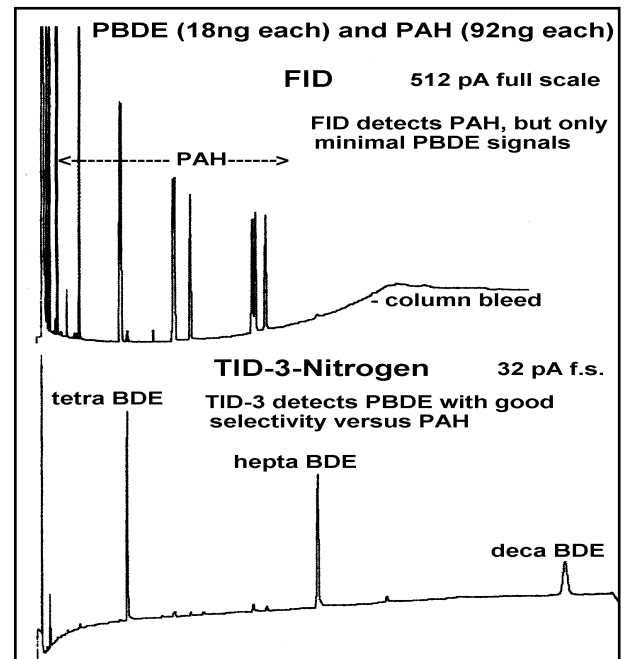
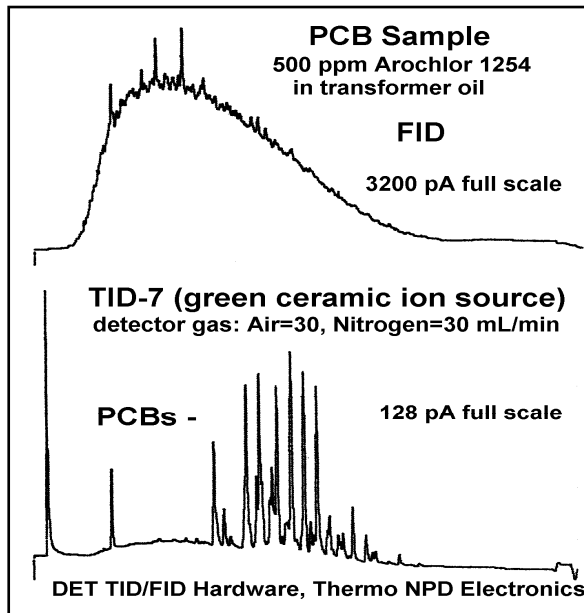
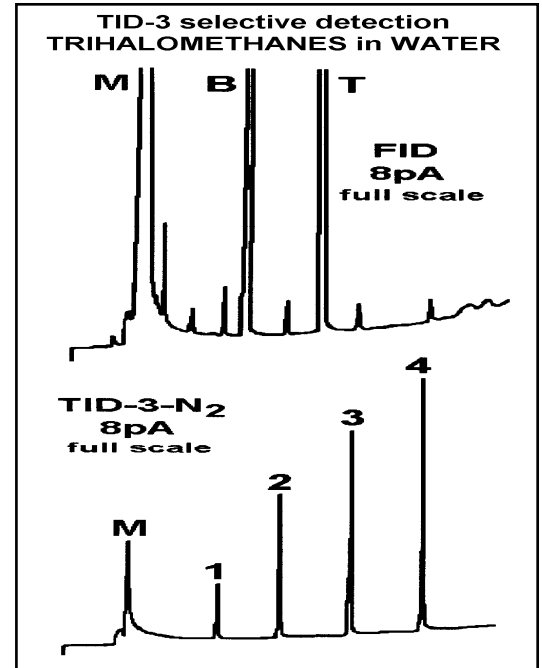
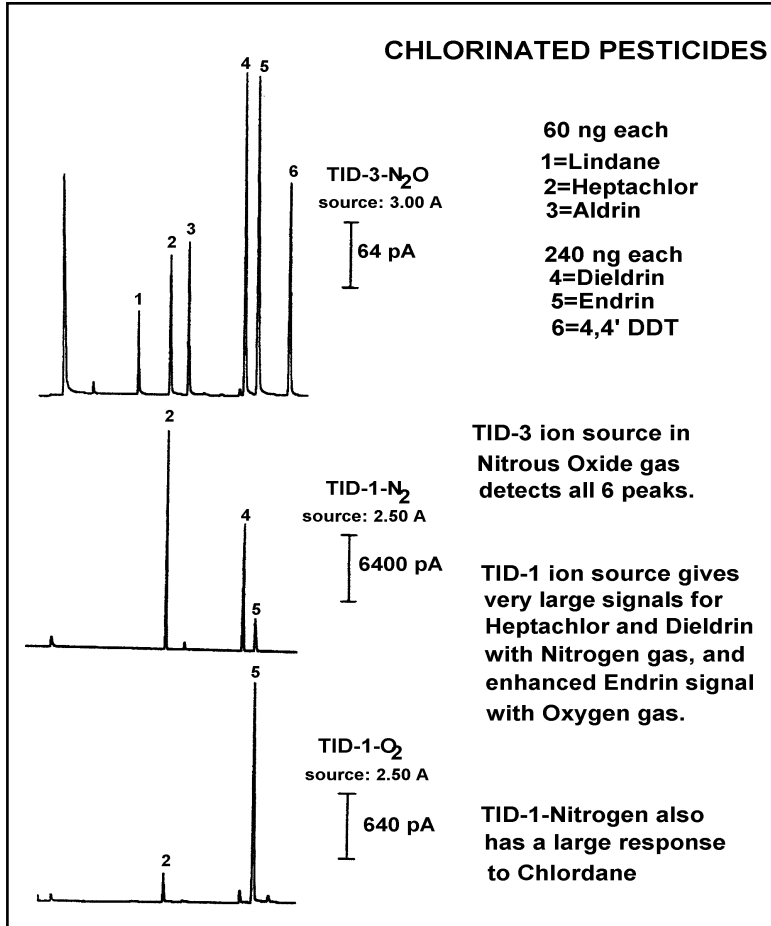
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TID-1 and newer version TID-10 ion sources can operate in either a detector gas environment of Nitrogen or Air and provide selectivity for many Oxygenated compounds. Air can be used to suppress responses from some Oxygenates relative to others – for example, Benzyl Salicylate vs. Ethyl Butyrate and a greatly reduced Acetone solvent. TID-1/TID-10 have exceptional sensitivity for Vanillin. Also, this detection is non-destructive, so distinguishing aromas of fragrance components can be sensed at the detector exit.

SRI GC with DET retrofit NPD/TID/FID hardware and ion sources powered with a stand-alone DET Current Supply polarized at – 45 V.

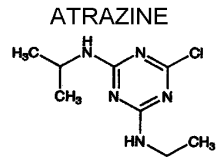
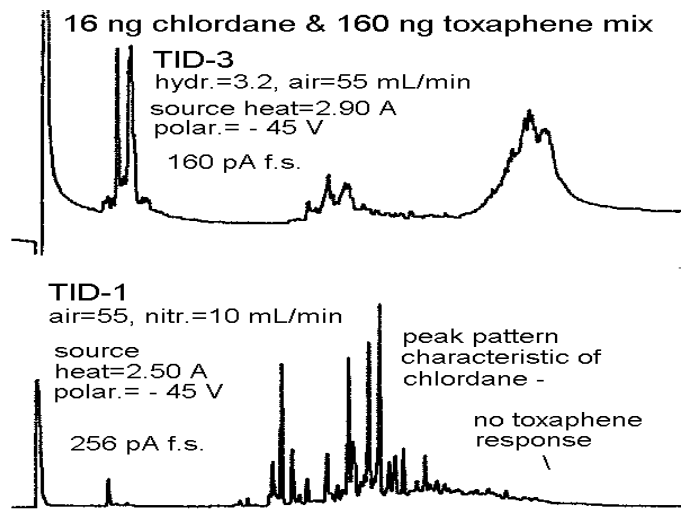
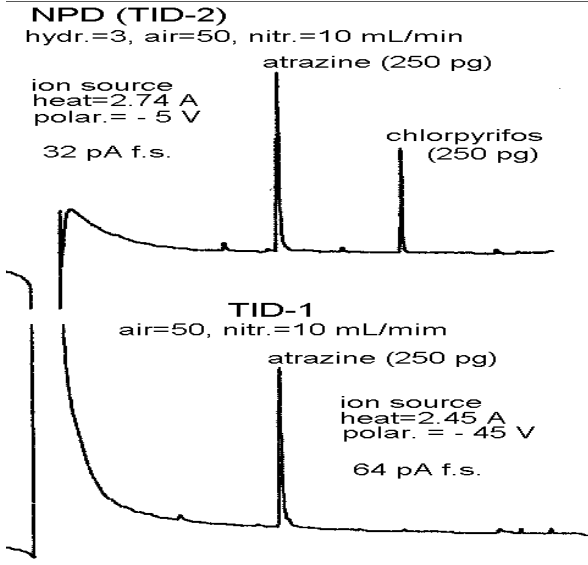
THERMIONIC SURFACE IONIZATION DETECTION FOR GC selectivity for HALOGENATED (Cl, Br, I) compounds



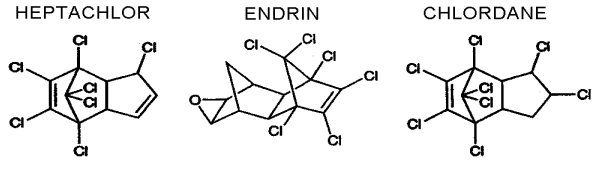
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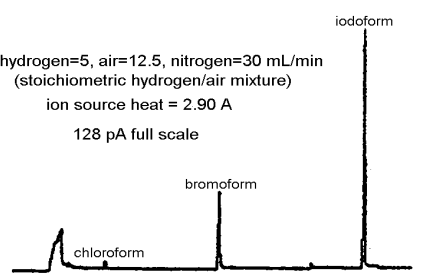
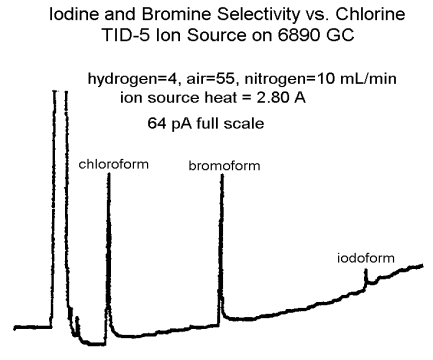
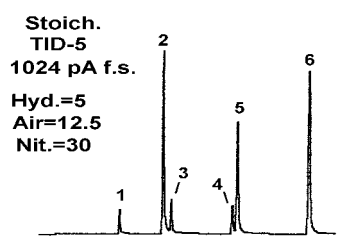
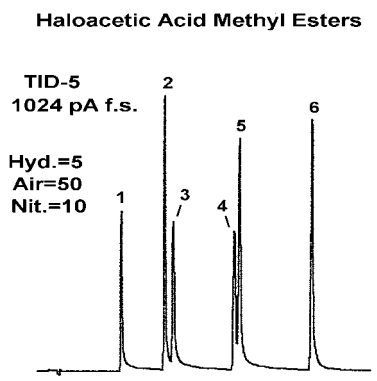
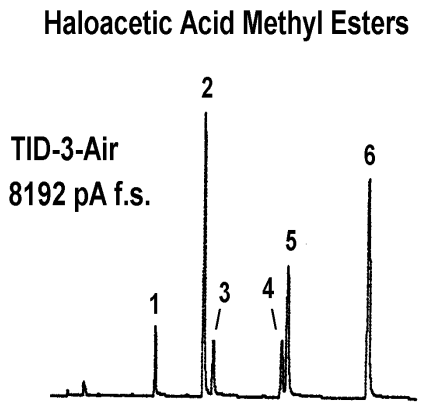
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TID-1-Air selectivity for Triazines is due to Cl atom in "para" location relative to other functionalities in the molecular structure of the sample compound.



TID-1(TID-10) has high sensitivity & selectivity for compounds like Heptachlor & Chlordane which have a common 6 Cl sub-structure.



- 1=Methyl Monochloroacetate
- 2=Methyl Monobromoacetate
- 3=Methyl Dichloroacetate,
- 4=Methyl Trichloroacetate,
- 5=Methyl Bromochloroacetate,
- 6=Methyl Dibromoacetate

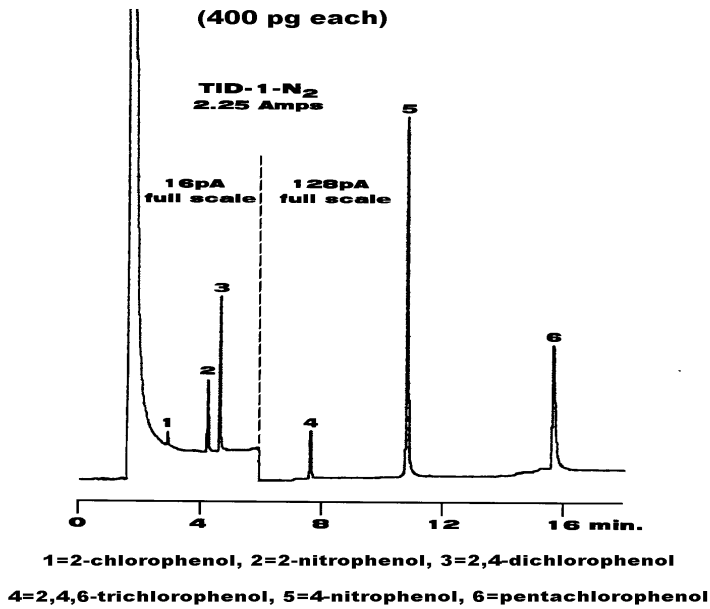
same sample as TID-3
 TID-5 & 2 different gas environments

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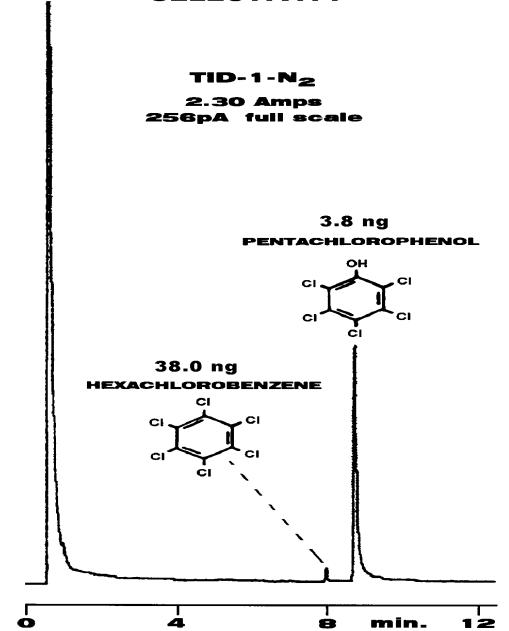
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CHLORO/NITRO-PHENOLS



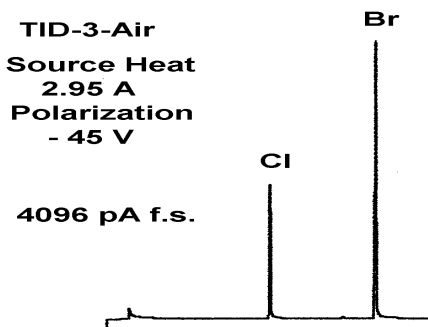
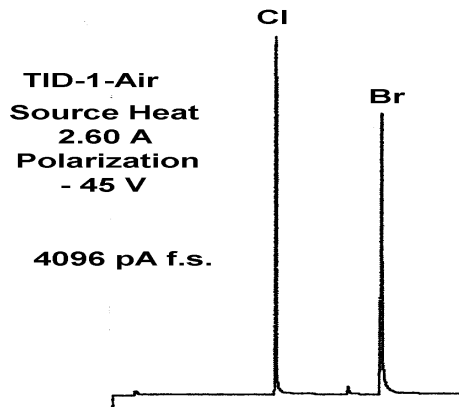
Cl and the NO₂ group are both strong electronegative functional groups, with NO₂ Phenols having larger TID-1 responses than comparable Cl Phenols.

PENTACHLOROPHENOL SELECTIVITY

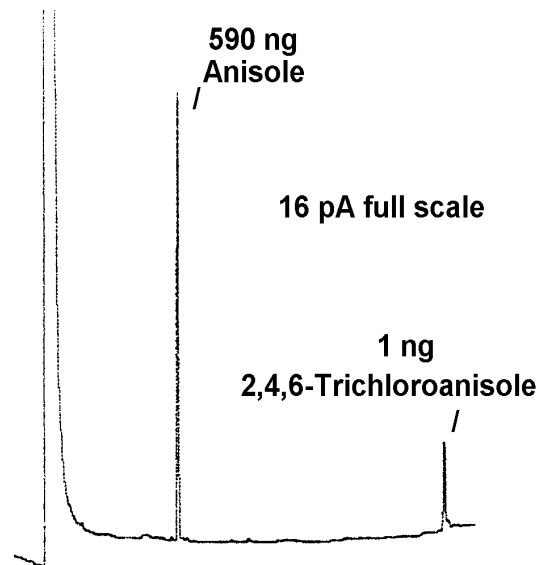


OH group instead of sixth Cl atom attached to ring gives much larger TID-1 response.

2,4,6 TRICHLORO/TRIBROMO PHENOLS



TID-1-Nitrogen

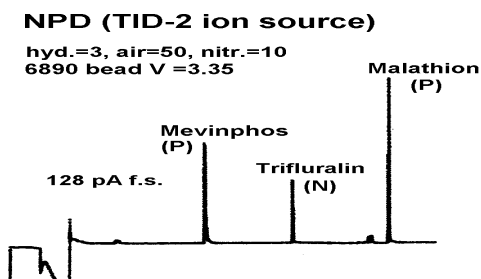
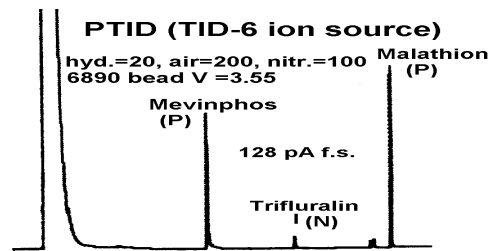
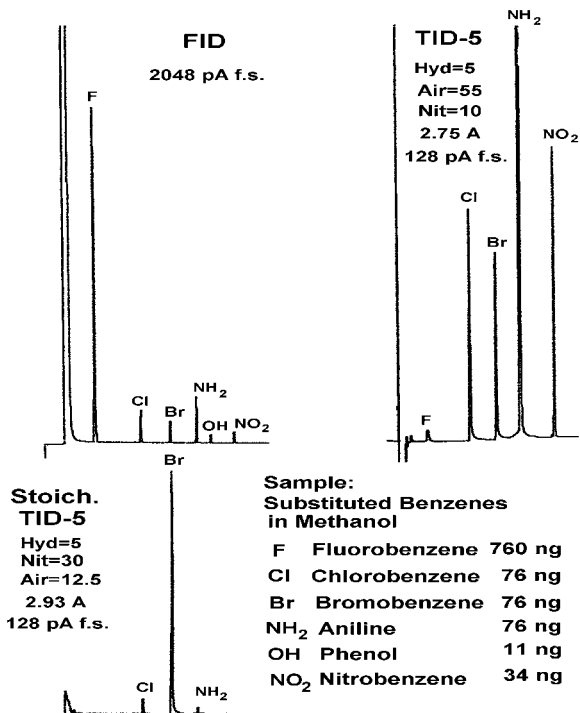


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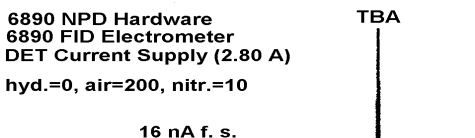
TID-5 Detection - 2 Versions



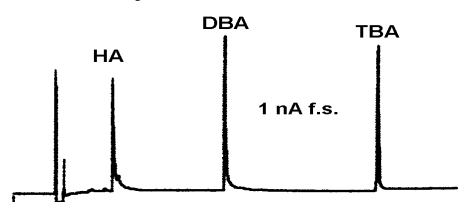
TID-6 ion source in 6890 NPD equipment with high H₂, Air, and Nitrogen flows provides P detection with N response suppressed compared to NPD.

TID-5 with stoichiometric mix of H₂ and Air provides selectivity for Br and I compounds versus Cl.

Thermionic Detection on 6890 GC Positive Ions on Bare Wire Probe

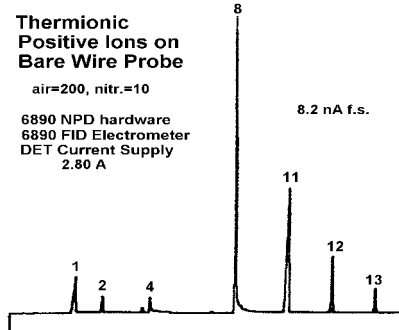
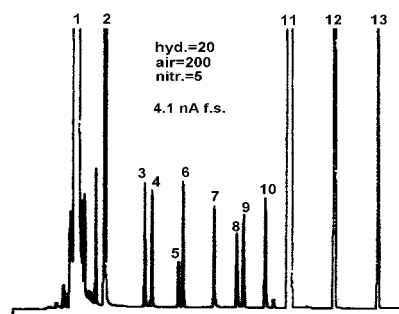


Negative Ions on TID-4 Ion Source



Sample: 7ng each of n-Hexylamine (HA), di-n-Butylamine (DBA), tri-n-Butylamine (TBA). Most DET modes of thermionic detection involve formation of negative ions. Positive ion thermionic detection is achieved with DET ionizing elements in Agilent 6890 NPD hardware, powered with a DET Current Supply, and signal measurement with an Agilent 6890 FID Electrometer.

Positive Ion Detection on 6890 GC Agilent AED #2 test sample FID with a Bare Wire Probe Ignitor



Positive ion FID and Thermionic detection on 6890 GC. Agilent AED #2 sample: peak 8=Triethyl Phosphate, 11=n-Dodecane, 12=n-Tridecane, 13=n-Tetradecane. Thermionic response to Hydrocarbons due to CH₂ functional group.

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CATALYTIC COMBUSTION IONIZATION DETECTION (CCID)

DET introduces a GC detection method that selectively ionizes METHYLENE (CH₂) functional groups in PETROLEUM, BIOFUEL, FAME, & TRIGLYCERIDE samples

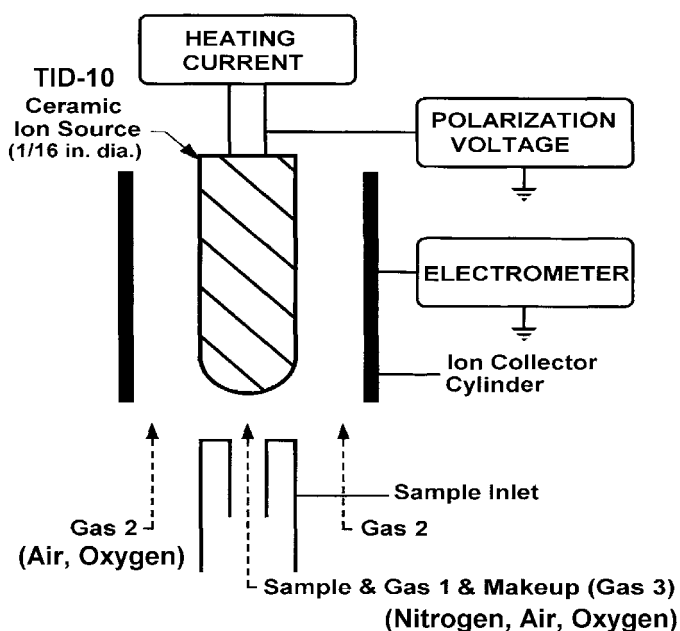
Equipment:

As in the schematic, a cylindrical ion source element is positioned on the axis of an ion collector cylinder, with detection gases 1, 2, and 3 being Air, Oxygen, or other Oxidizing gas. A TID-1 or TID-10 ceramic ion source is electrically heated to 300 - 400°C, and polarized at a negative voltage relative to the ion collector.

Principle of Detection:

As they elute sequentially from the GC column, sample compounds corresponding to a high concentration of a Hydrocarbon or other Organic provide the fuel to momentarily ignite a burst of flame ionization as each compound impacts the hot catalytically active ceramic. Compounds containing chains of Methylene functional groups are selectively detected versus other compounds.

CATALYTIC COMBUSTION IONIZATION

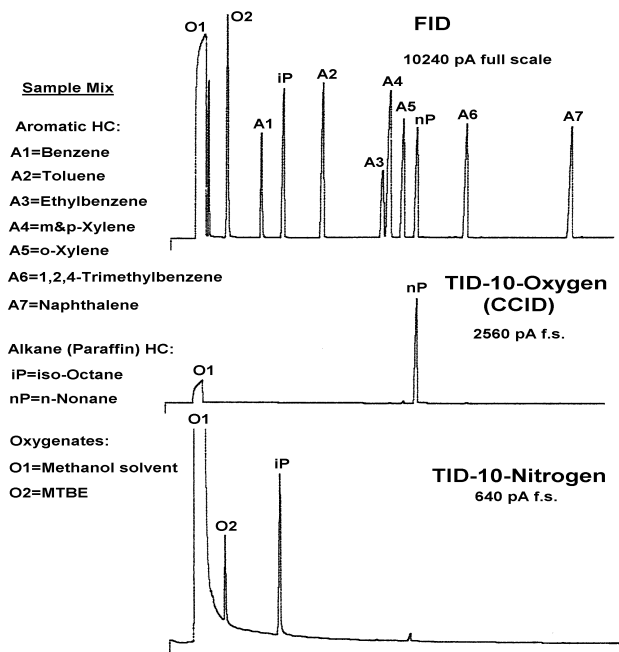


Important Consequences of this Chemical Detection

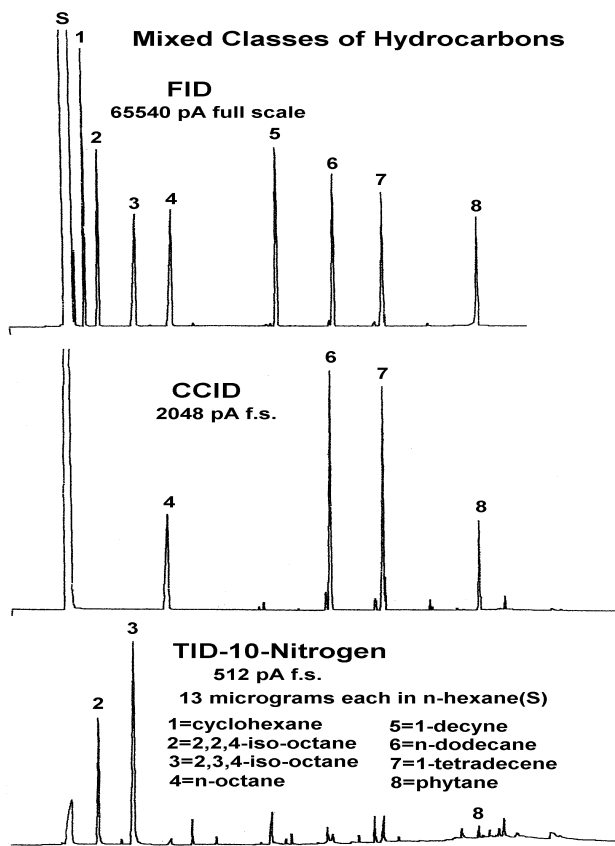
1. Demonstrates that high temperature oxidation of CH₂ groups is a primary process contributing to the combustion ignition of Petroleum, Biofuel, and FAME constituents.
2. The temperature required for ignition of fuel combustion is lowered with increased catalytic activity of the ceramic. Method allows combustion study using many different catalysts.
3. GC chromatograms of different fuel samples provide fingerprint patterns showing the individual compounds of each sample which are most easily ignited in combustion.
4. Compounds with saturated Carbon bonds ignite in combustion more easily than compounds with Carbon double or triple bonds, and the magnitude of the difference depends on the concentration of Oxygen in the detector gases. Similarly, linear chain compounds are more readily ignited than branched chain compounds.
5. Aromatics or Naphthenes are NOT easily ignited in combustion by this technique.

CATALYTIC COMBUSTION IONIZATION DETECTION (CCID) selective detection of Linear Chain Alkanes vs. other Hydrocarbons

TID & CCID DISCRIMINATION VS. AROMATIC HC



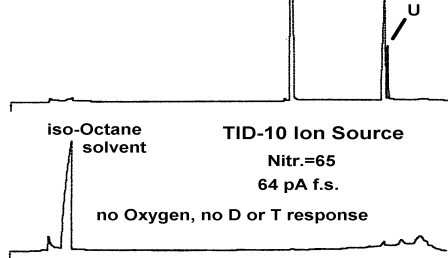
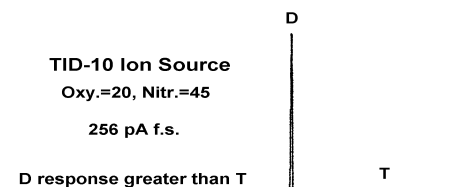
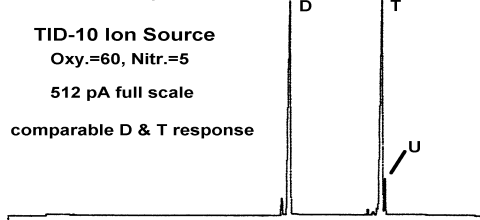
**CCID and TID DO NOT RESPOND to Aromatics (A1 - A7).
 CCID responds to Linear Alkane (nP), but not Branched Alkane (iP). TID responds to (iP) and Oxygenates.**



CCID responds to multiple CH₂ 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 CH₂ groups.

Alkane vs. Alkene Response Comparison

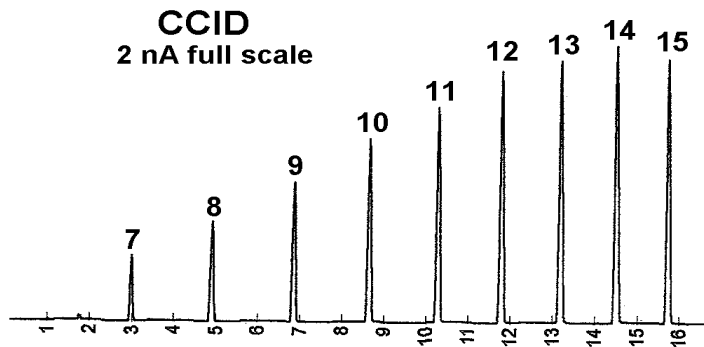
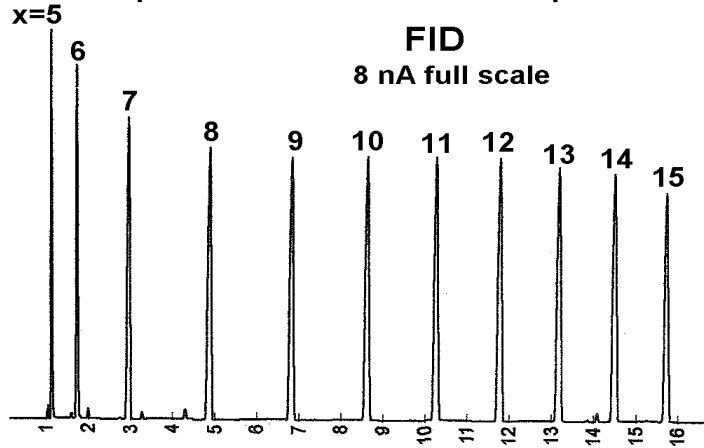
1% each, n-dodecane (D) and 1-tetradecene (T)
 (U=impurity)



**CCID (TID-10 ion source in Oxygen gas environment)
 Selective Detection of Methylene (CH₂) 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 CH₂ groups. Compounds with a Carbon triple bond (Alkynes) are similarly NOT very responsive to CCID detection even though they may contain many CH₂ groups. CCID selectivity dependence on compound bonding details provides unprecedented new chemical detection capability for GC.

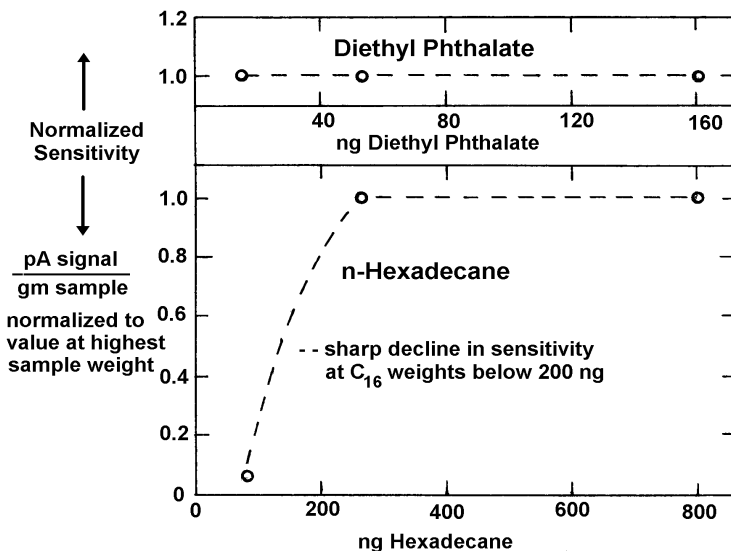
Mixture of n-Paraffins (Linear Chain Alkanes)
 equal amounts of each C_x compound



Comparison of CCID vs. FID responses to a mixture of n-Paraffins illustrates that CCID response gets bigger and bigger with increasing numbers of Methylene (CH₂) groups in the sample compound. When applied to a complex Petroleum sample, CCID provides a relative magnification of the longer chain sample constituents.

The TID-10 (and its predecessor TID-1) ion source used in CCID detection also responds to certain Heteroatom compounds (eg., Oxygenates) by a process of Thermionic Surface Ionization involving direct extraction of negative charge from the ion source. By contrast, CCID detection involves combustion ignition as the sample compound fuels a momentary burst of flame ionization when impacting the heated ion source. Therefore, there is a threshold sample amount below which there is not sufficient amount of sample to fuel CCID response.

TID-1-AIR

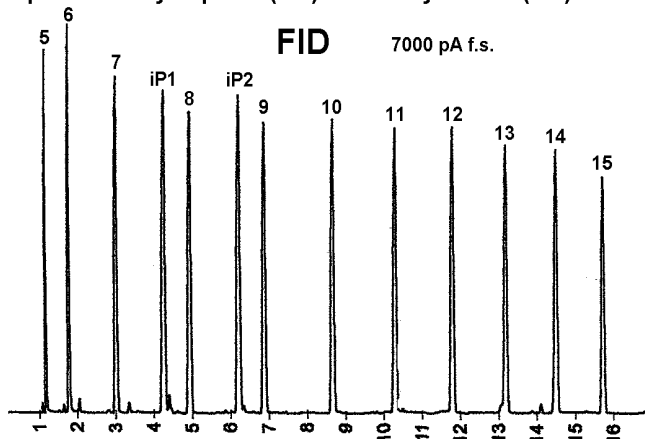


This is illustrated in the graph comparing responses of n-Hexadecane and Diethyl Phthalate. The CCID response threshold depends on the number of Methylene groups in the sample compound, as well as on the concentration of Oxygen in the detector gases and the surface temperature of the ion source. The threshold moves toward lower sample amounts with increasing numbers of Methylene groups, Oxygen concentration, and ion source temperature.

Sensitivity vs. Sample Amount Graph - linear response indicated by a horizontal line.

CCID Response Differences Paraffins vs. Iso-Paraffins

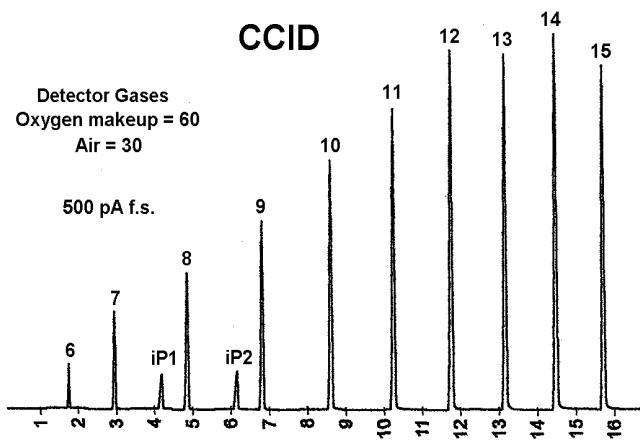
approx. equal mix of C5 - C15 n-Paraffins
plus 2-Methylheptane (iP1) & 4-Methyloctane (iP2)



CCID

Detector Gases
Oxygen makeup = 60
Air = 30

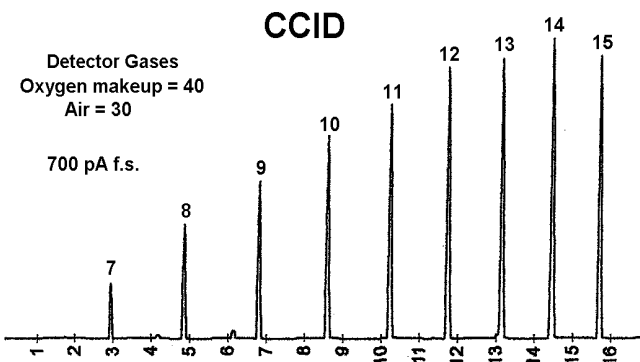
500 pA f.s.



CCID

Detector Gases
Oxygen makeup = 40
Air = 30

700 pA f.s.



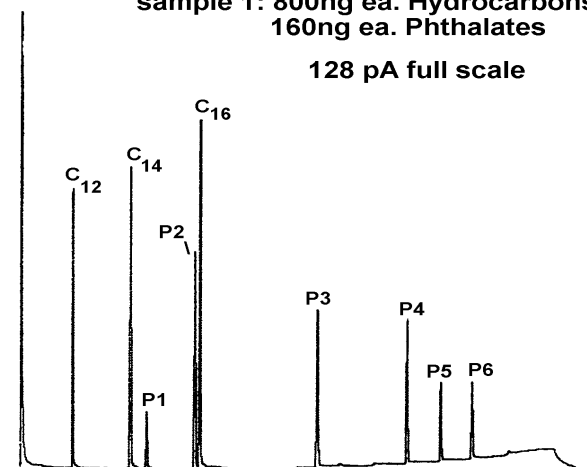
CCID responds to compounds containing chains of Methylene (CH_2) groups. The response is diminished when the compound also contains a branched functional group. Relative response of iso-Paraffins vs. n-Paraffins improves with increasing Oxygen concentration in the detector gases.

CCID Response Threshold vs. TID Surface Ionization (effect of sample dilution)

TID-1-AIR

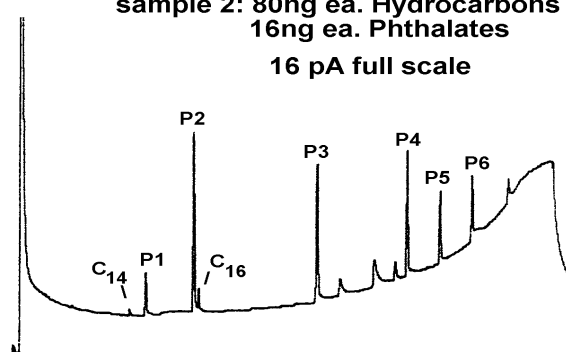
sample 1: 800ng ea. Hydrocarbons
160ng ea. Phthalates

128 pA full scale



sample 2: 80ng ea. Hydrocarbons
16ng ea. Phthalates

16 pA full scale



This sample - peaks labeled "C" are Linear Chain Alkanes while peaks labeled "P" are Phthalates.

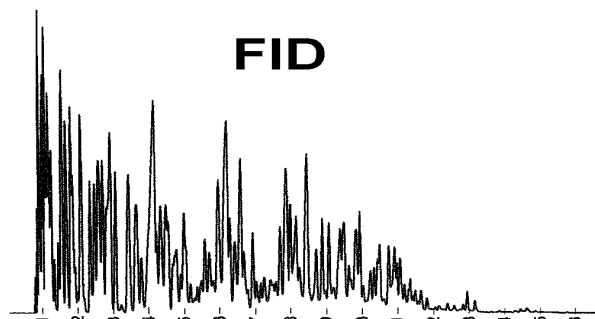
CCID response involves a TID-10 (or previous TID-1) ion source operated in a detector environment containing Oxygen. The same ion source also produces selective responses to some Heteroatom compounds due to a process of direct Thermionic Surface Ionization. For sample analyses containing both CCID and TID peaks, the CCID peaks disappear with sample dilution below the CCID response threshold. TID responses generally can prevail to much lower sample component amounts.

DET

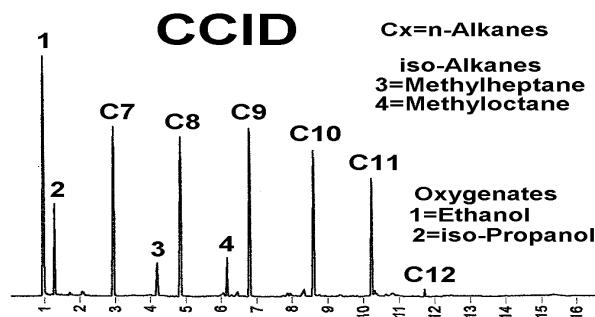
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GASOLINE ANALYSIS



FID



CCID

Cx=n-Alkanes

iso-Alkanes

3=Methylheptane

4=Methyloctane

Oxygenates

1=Ethanol

2=iso-Propanol

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 (CH₂) 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.

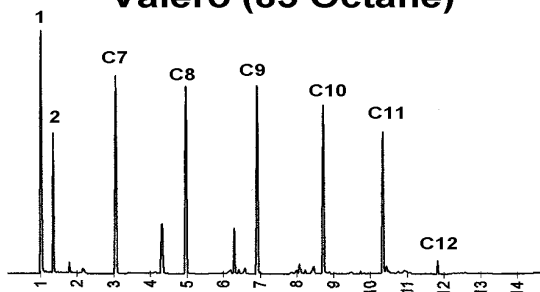
The catalytically active TID-10 ceramic ion source used in CCID also responds to Oxygenated constituents via a Thermionic Surface Ionization process (TID) which is different than CCID. Hence, the gasoline chromatograms illustrated here are comprised of both CCID and TID selective responses. As shown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

Same chromatographic conditions were used for all data.

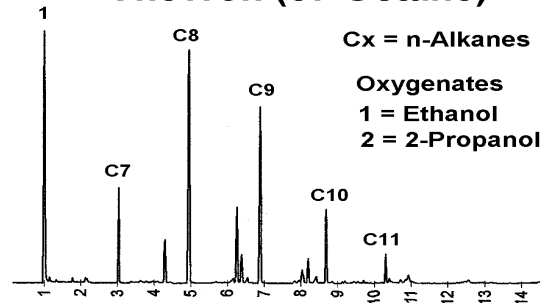
DET retrofit on Thermo Ultra GC, Air=30, O₂ makeup=50mL/min, TID-10 ion source heat=2.45A, polariz.= - 90V. 0.5: L gasoline, 30m x 0.53mm DB1, He=8, 40-60°C at 5°C/min, 60-190°C at 10°C/min, detector=250°C.

CCID ANALYSIS OF GASOLINES

Valero (85 Octane)



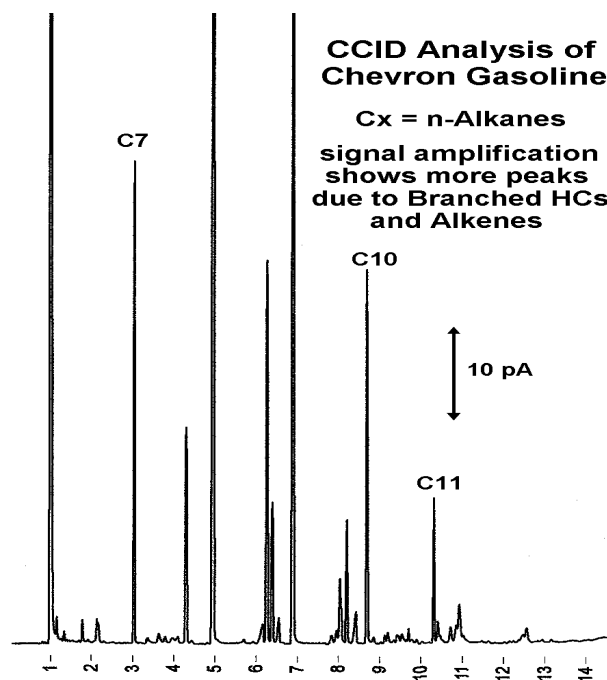
Chevron (87 Octane)



CCID Analysis of Chevron Gasoline

Cx = n-Alkanes

signal amplification shows more peaks due to Branched HCs and Alkenes



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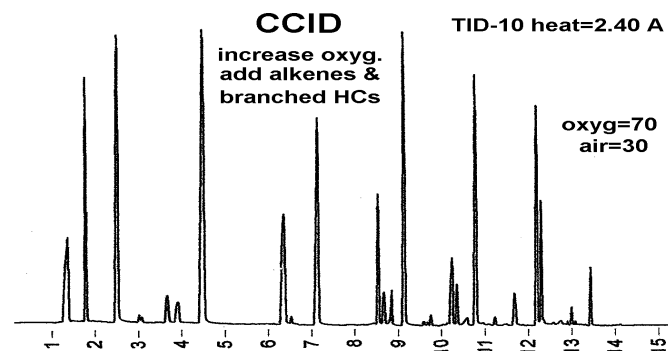
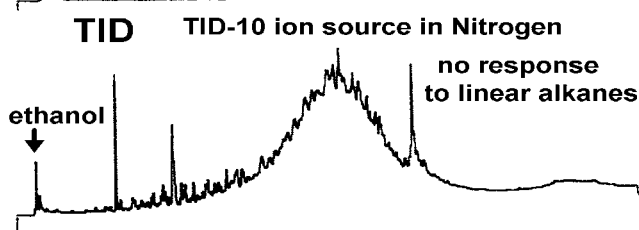
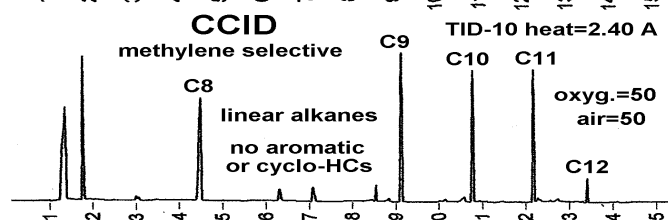
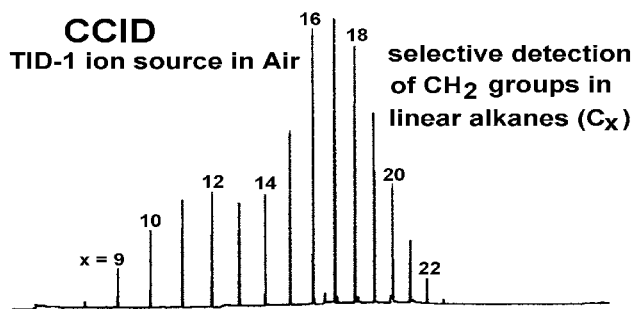
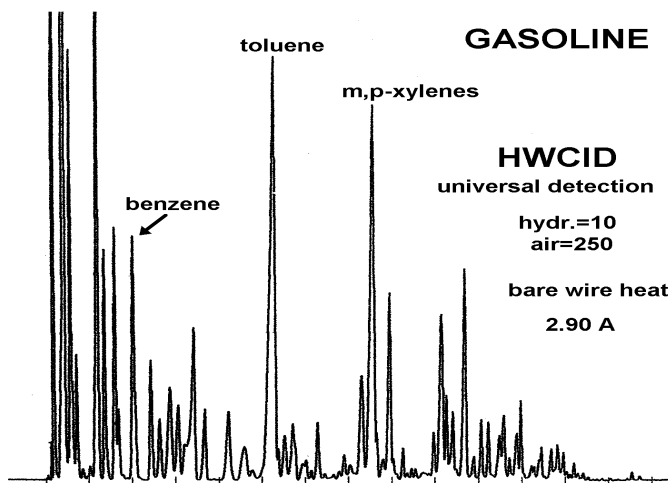
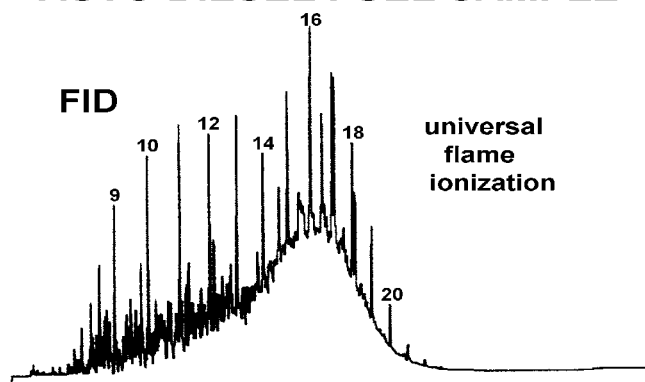
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CCID & TID ANALYSES OF SELECTIVE COMPONENTS OF PETROLEUM FUELS

VALERO AUTO DIESEL

VALERO PREMIUM GASOLINE

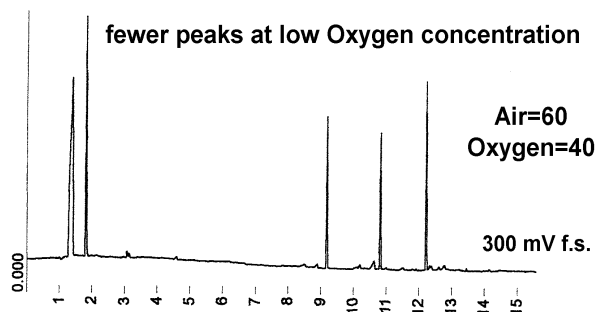
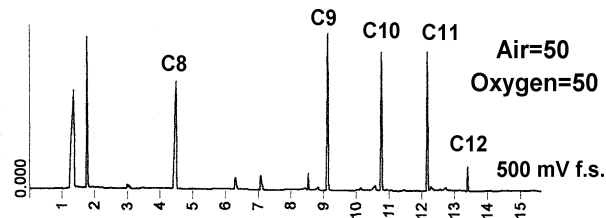
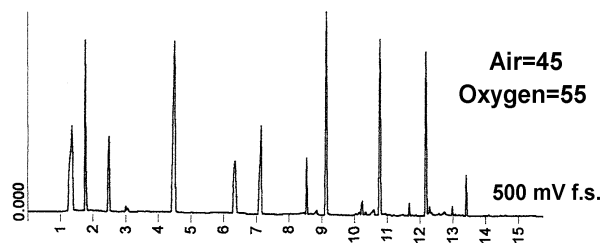
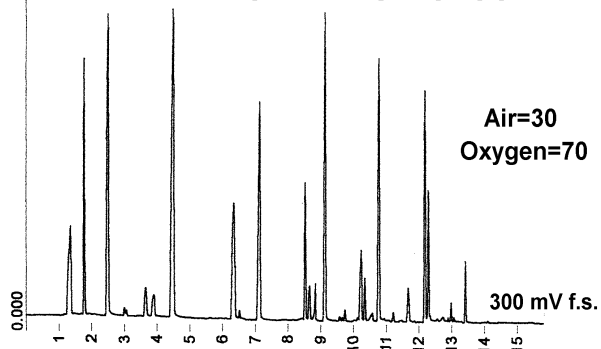
AUTO DIESEL FUEL SAMPLE



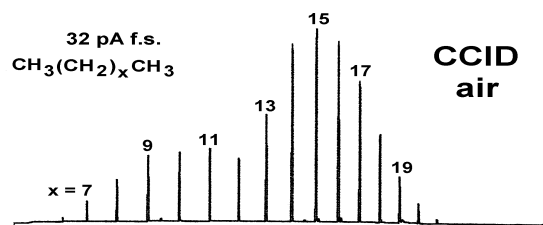
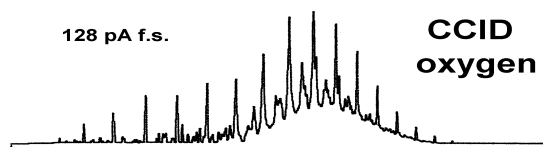
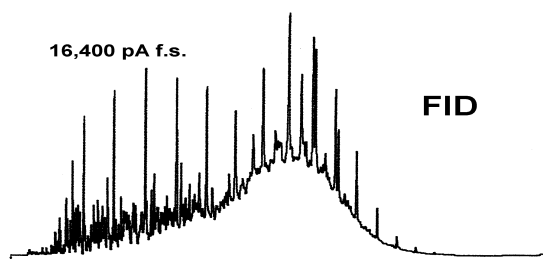
Compared to the FID chromatogram, CCID detection with an oxidizing Air detector gas environment singles out just the Linear Alkane Hydrocarbon constituents, while TID detection with an inert Nitrogen gas reveals Hydrocarbon constituents other than Linear Alkanes and Aromatics, plus Heteroatom constituents like Oxygenates. Neither TID or CCID detect Aromatic Hydrocarbons.

HWCID (Hot Wire Combustion Ionization) provides universal FID-like detection with enhanced Aromatic responses. CCID with a low concentration of Oxygen in the detector gas reveals the Linear Alkane constituents of the gasoline, while increasing the Oxygen concentration in the detector gas adds peaks due to Alkene and Branched Hydrocarbon constituents.

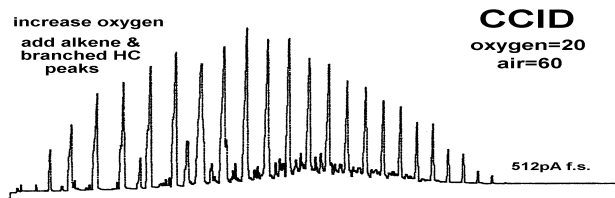
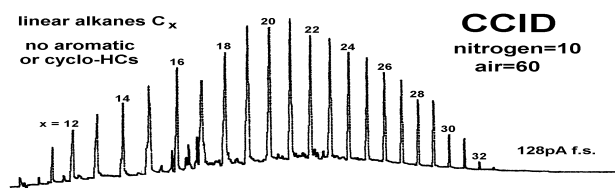
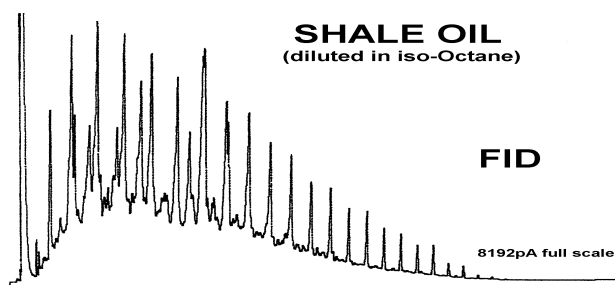
CCID - VALERO PREMIUM GASOLINE



AUTO DIESEL #2 FUEL



SHALE OIL (diluted in iso-Octane)

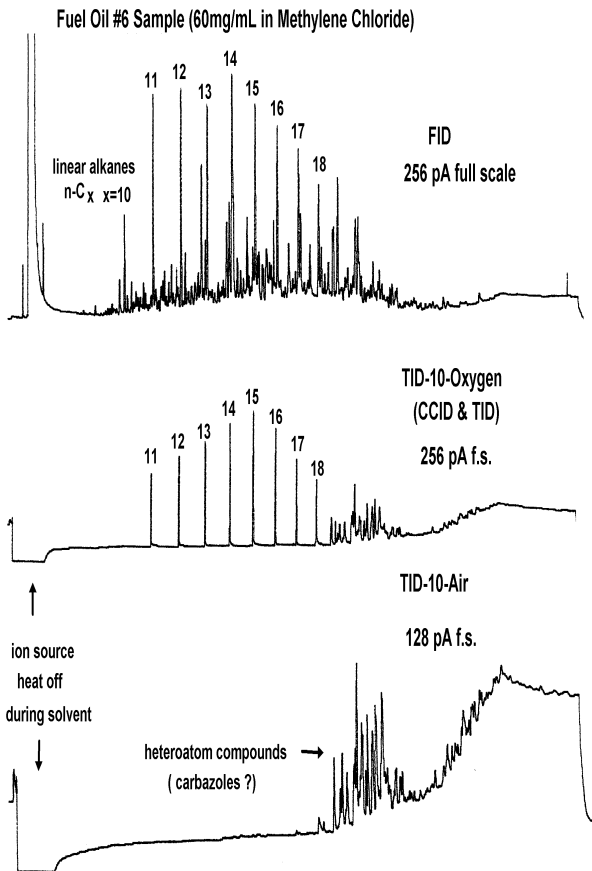


These data illustrate how judicious increases in the detector Oxygen concentration can further characterize CCID chromatograms of Gasoline samples by adding Alkene (Olefin) and Branched Hydrocarbon (iso-Paraffin) peaks to the Linear Alkanes (n-Paraffins) of the sample, while still having negligible response to the Aromatic or Cyclo-Hydrocarbon (Naphthene) components of the sample. In the "PIANO" analysis terminology for petroleum samples, CCID at low Oxygen concentration provides a "P" type of analysis, and a "PIO" type of analysis at higher Oxygen.

Compared to FID analyses of Petroleum, CCID reveals Paraffin and Olefin constituents without the Aromatics and Naphthenes that elute earlier.

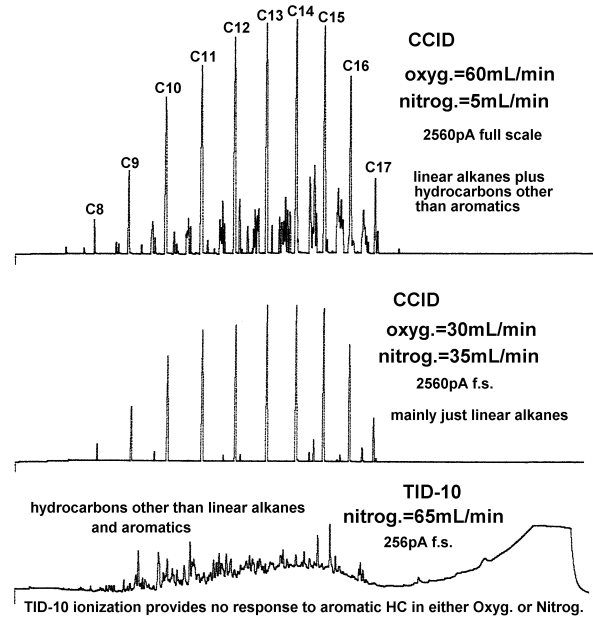
CATALYTIC COMBUSTION IONIZATION (CCID) & THERMIONIC SURFACE IONIZATION (TID)

inexpensive detection of selected constituents in complex Petroleum samples
(CCID/TID are easy alterations of NPD equipment on Thermo, Agilent, Varian/Bruker, and SRI GC models)

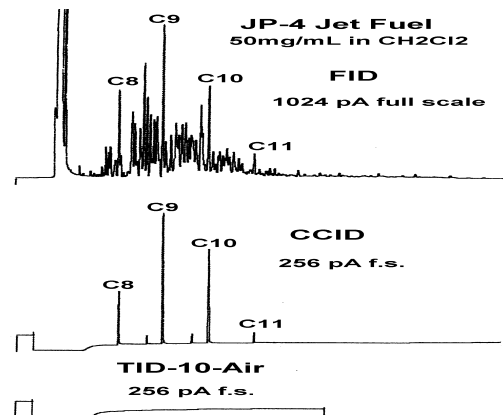


DILUTED FUEL OIL sample - TID-10 ion source in an O_2 gas environment provides CCID selective detection of Linear Alkanes plus late retention time peaks corresponding to TID selective detection of heteroatom Carbazole compounds which contain the Pyrrole functional group - decreasing the O_2 by change to Air detector gas removes the CCID Linear Alkane peaks and leaves only the TID heteroatom peaks - CCID detection requires that the concentration of the sample compound is high enough to fuel a momentary burst of flame ionization as the compound impacts the heated, catalytically active TID-10 ceramic surface - the sample concentration at which this threshold of CCID detection occurs depends on the available O_2 concentration and the temperature of the ceramic - higher O_2 and surface temperature allow CCID detection to occur at a lower sample concentration.

Undiluted Kerosene - CCID/TID Selectivity vs. Oxygen/Nitrogen



UNDILUTED (NEAT) KEROSENE - CCID selectivity can be varied by adjusting the O_2 concentration in the detector gases - at low O_2 , just Linear Alkanes are detected - at higher O_2 , additional peaks from Alkenes and Branched Hydrocarbons appear - TID peaks with just N_2 have not yet been identified.

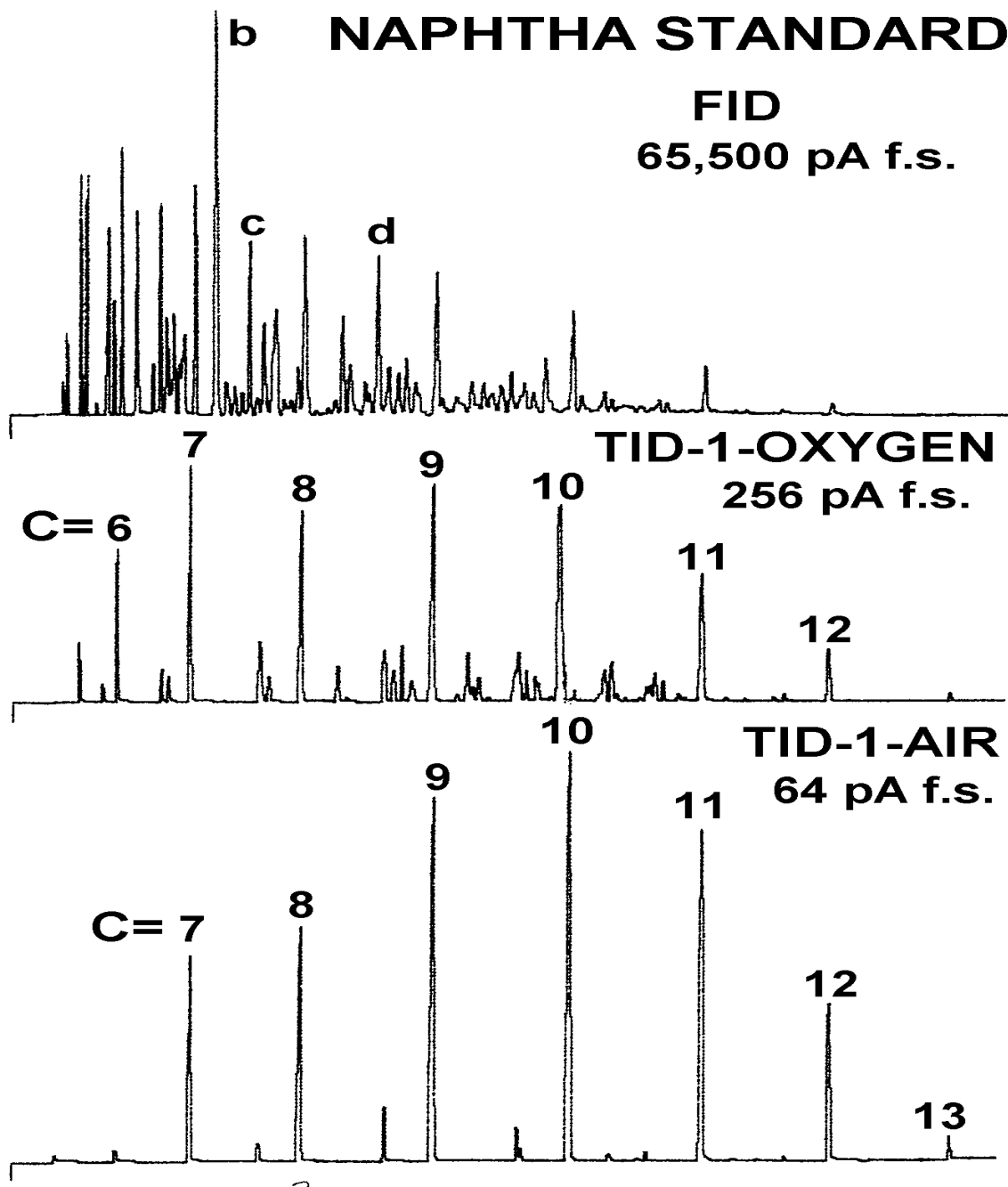


DILUTED JET FUEL - CCID with O_2 detects Linear Alkanes - decrease in O_2 with change to Air detector gas provides no response for this diluted sample.

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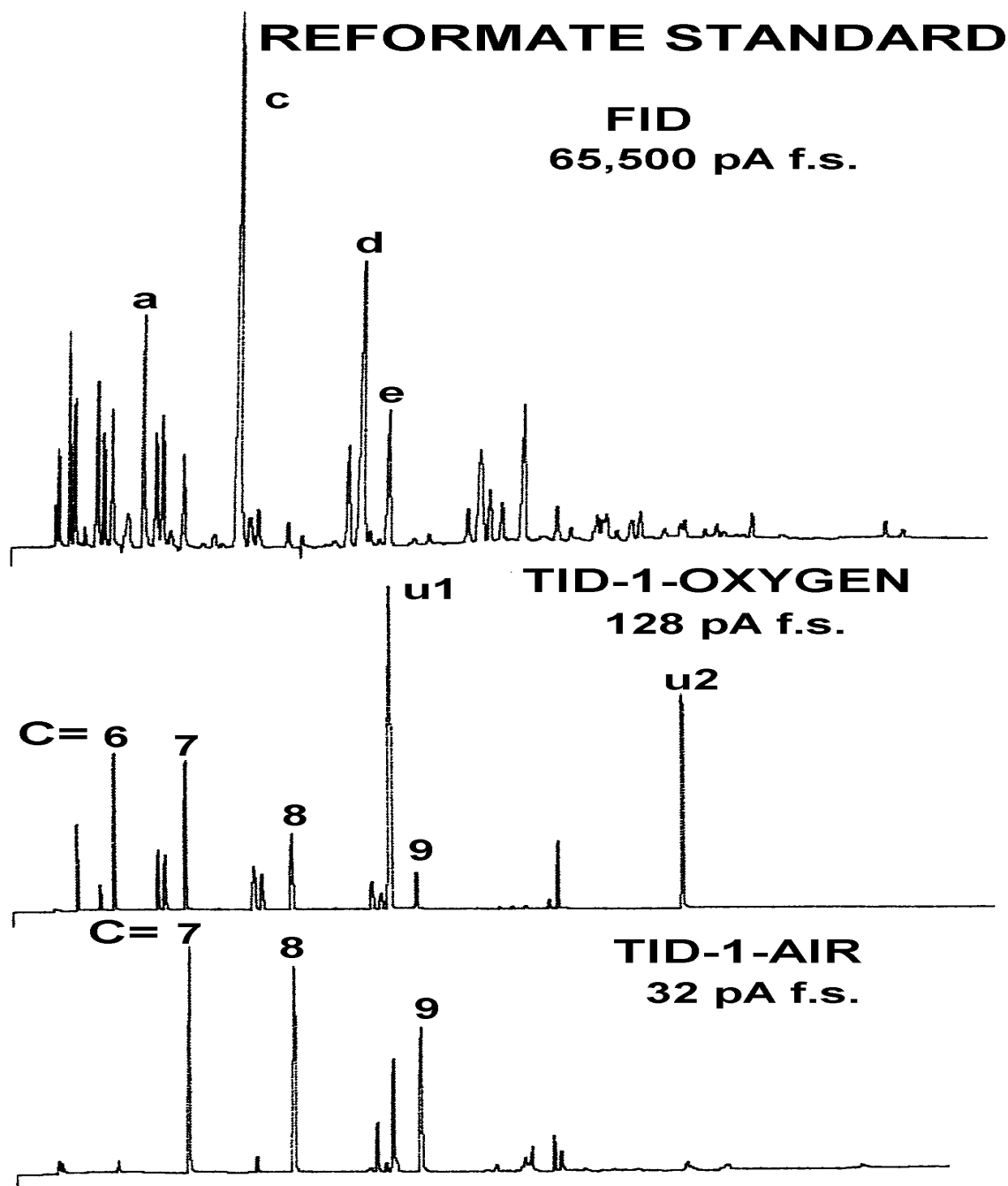


Agilent 6890 GC. TID-1 ion source in 6890 NPD hardware with DET Current Supply for ion source power. C=x indicates Carbon number in linear Alkanes; b = Methyl-cyclohexane & cis-1,2-Dimethylcyclopentane; c = Toluene; d = m-Xylene & p-Xylene. TID-10 source is similar.

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Agilent 6890 GC. TID-1 ion source in 6890 NPD hardware with DET Current Supply for ion source power. C=x indicates Carbon number in Linear Alkanes; a = Benzene; c = Toluene; d = m-Xylene & p-Xylene; e = o-Xylene; u1 & u2 = unidentified components (likely heteroatoms).

DET

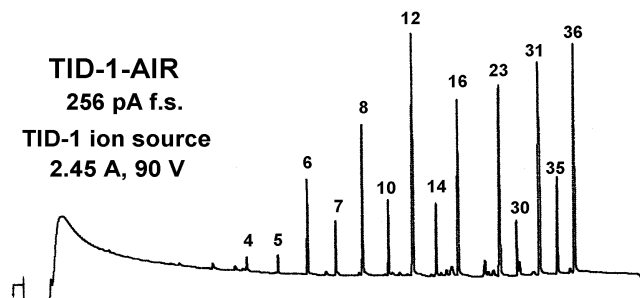
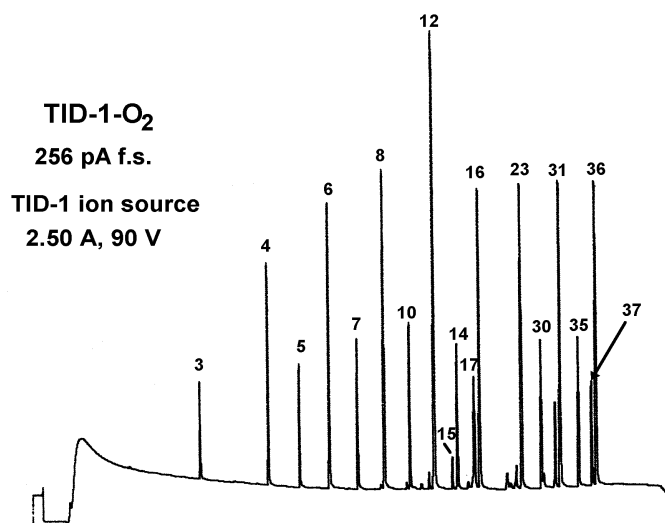
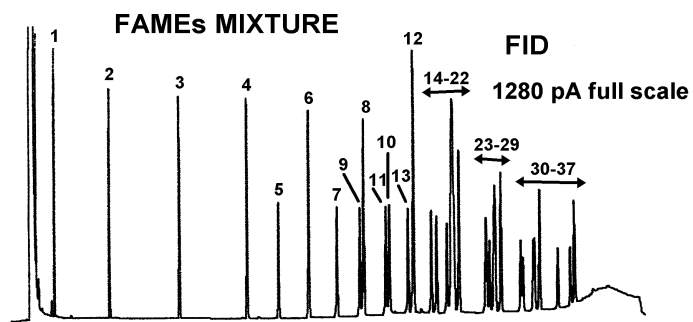
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CCID Detection of FAMES

FAMES SAMPLE:

- 1.) Butyric (C4:0), 4wt%;
- 2.) Caproic (C6:0), 4wt%;
- 3.) Caprylic (C8:0), 4wt%;
- 4.) Capric (C10:0), 4wt%;
- 5.) Undecanoic (C11:0), 2wt%;
- 6.) Lauric (C12:0), 4wt%;
- 7.) Tridecanoic (C13:0), 2wt%;
- 8.) Myristic (C14:0), 4wt%;
- 9.) Myristoleic (C14:1), 2wt%;
- 10.) Pentadecanoic (C15:0), 2wt%;
- 11.) cis-10-Pentadecenoic (C15:1), 2wt%;
- 12.) Palmitic (C16:0), 6wt%;
- 13.) Palmitoleic (C16:1), 2wt%;
- 14.) Heptadecanoic (C17:0), 2wt%;
- 15.) cis-10-Heptadecenoic (C17:1), 2wt%;
- 16.) Stearic (C18:0), 4wt%;
- 17.) Oleic (C18:1n9c), 4wt%;
- 18.) Elaidic (C18:1n9t), 2wt%;
- 19.) Linoleic (C18:2n6c), 2wt%;
- 20.) Linolelaidic (C18:2n6t), 2wt%;
- 21.) γ -Linolenic (C18:3n6), 2wt%;
- 22.) α -Linolenic (C18:3n3), 2wt%;
- 23.) Arachidic (C20:0), 4wt%;
- 24.) cis-11-Eicosenoic (C20:1n9), 2wt%;
- 25.) cis-11,14-Eicosadienoic (C20:2), 2wt%;
- 26.) cis-8,11,14-Eicosatrienoic (C20:3n6), 2wt%;
- 27.) cis-11,14,17-Eicosatrienoic (C20:3n3), 2wt%;
- 28.) Arachidonic (C20:4n6), 2wt%;
- 29.) cis-5,8,11,14,17 Eicosapentaenoic (C20:5n3), 2wt%;
- 30.) Heneicosanoic (C21:0), 2wt%;
- 31.) Behenic (C22:0), 4wt%;
- 32.) Erucic (C22:1n9), 2wt%;
- 33.) cis-13,16-Docosadienoic (C22:2), 2wt%;
- 34.) cis-4,7,10,13,16,19-Docosahexaenoic (C22:6n3), 2wt%;
- 35.) Tricosanoic (C23:0), 2wt%;
- 36.) Lignoceric (C24:0), 4wt%;
- 37.) Nervonic (C24:1n9), 2wt%.



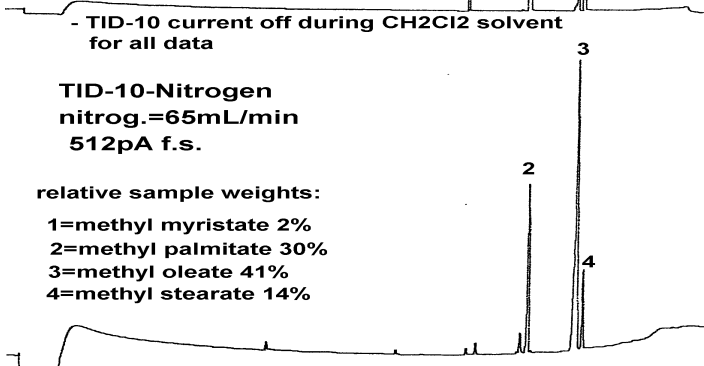
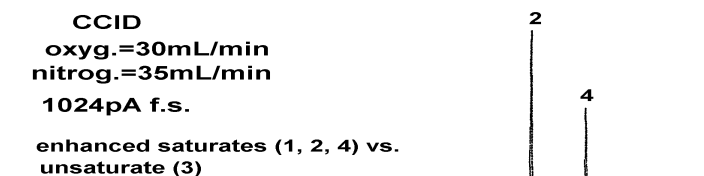
CCID (TID-1) detection of chains of CH₂ groups in Fatty Acid Methyl Esters (FAMES). Sample contained both Saturated and Unsaturated FAMES. TID-1-Air detected only the Saturates. TID-1-Oxygen added Mono-Unsaturate peaks but no Poly-Unsaturates. Same response with TID-10.

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.

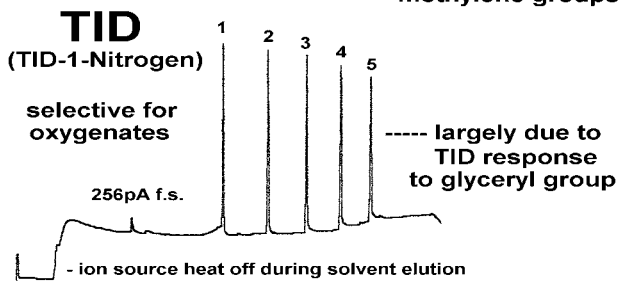
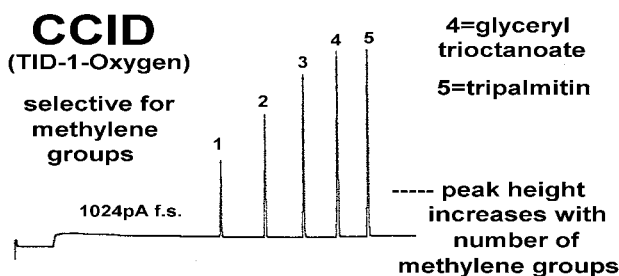
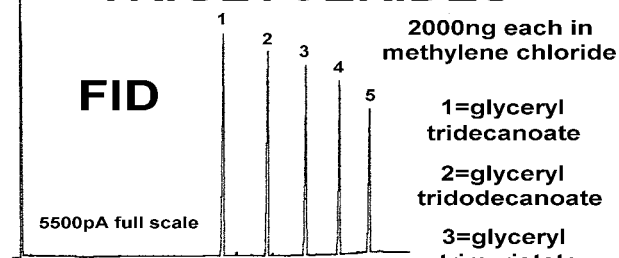
CCID/TID Responses vs. Oxygen/Nitrogen

Mixture of FAME 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.

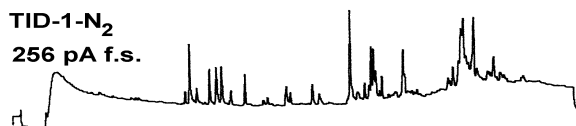
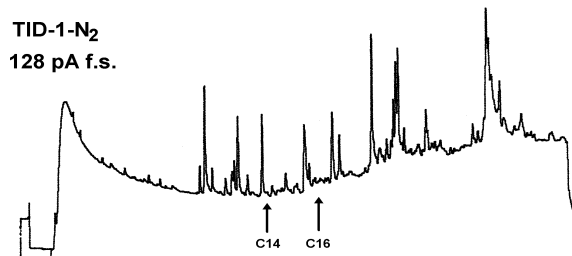
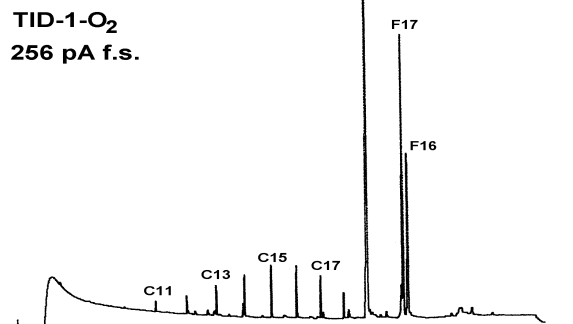
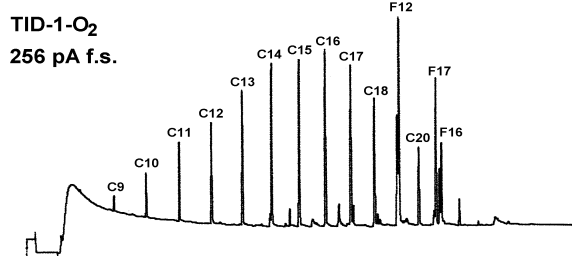
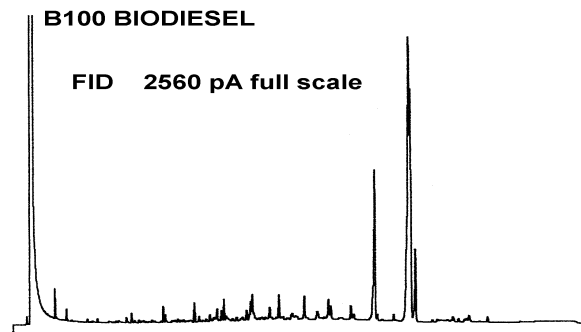
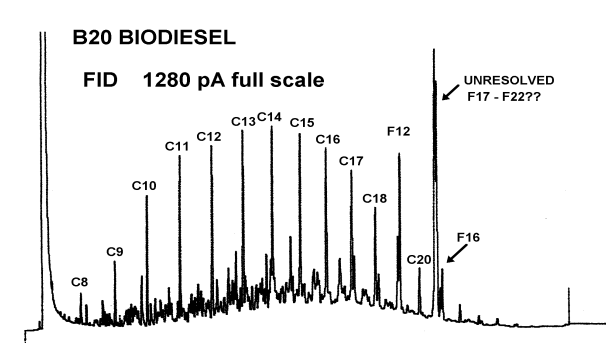
TRIGLYCERIDES



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.

CATALYTIC COMBUSTION IONIZATION (TID-1-O₂) selective detection of Linear Alkanes & FAMES in Biofuels

(TID-1 & TID-10 are equivalent ceramic ion source choices for Catalytic Combustion Ionization)



20mg/mL B20 Biodiesel diluted in Methylene Chloride.

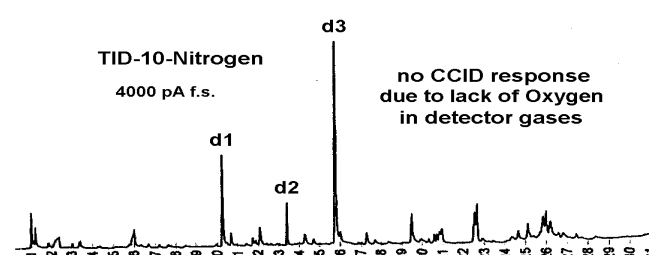
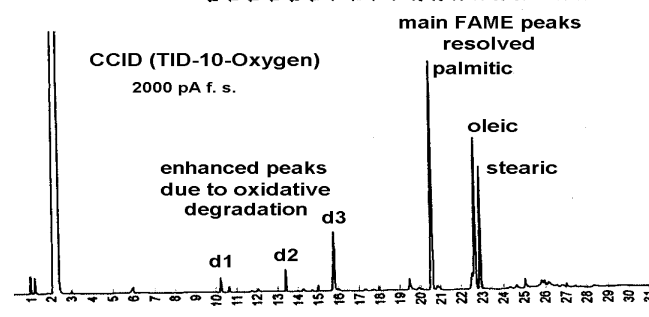
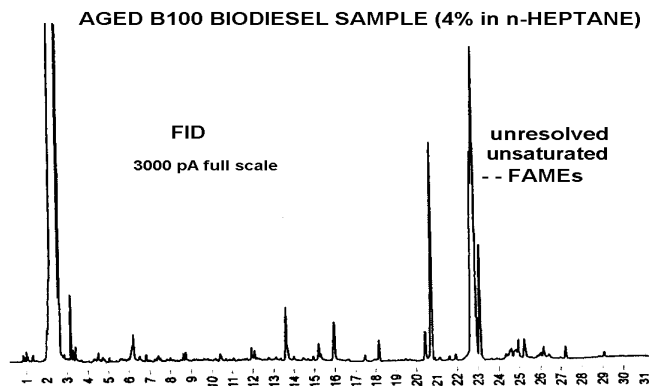
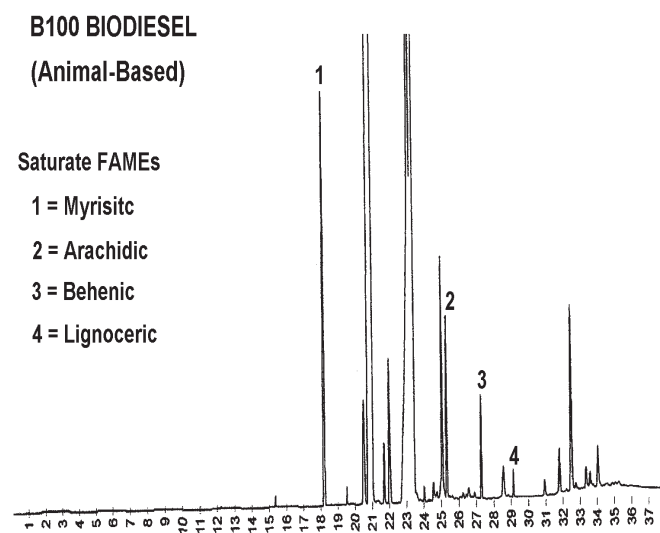
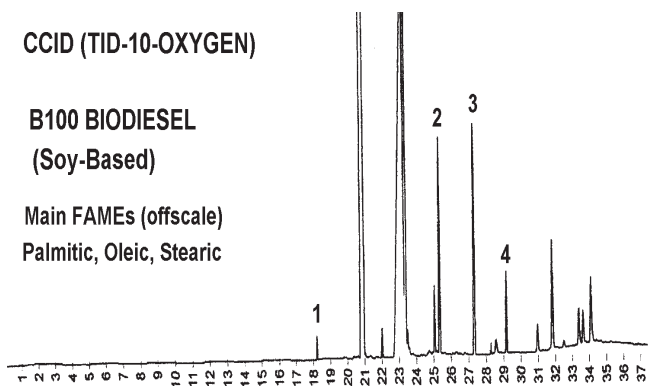
20mg/mL B100 Biodiesel in Methylene Chloride.

Catalytic Combustion Ionization Detection (CCID) provides selective detection of chains of Methylene (CH₂) groups in Linear Alkane (peaks labeled C_x) and FAME (peaks labeled F_x) constituents of the sample. Enhanced sensitivity of saturated versus unsaturated FAMES diminishes interfering responses from some unsaturates and allows clear definition of the 3 major FAME components - Palmitic Acid (F₁₂), Oleic Acid (F₁₇), and Stearic Acid (F₁₆) Methyl Esters. CCID requires the presence of O₂ in the detector gases. Using the same ion source and changing to an inert N₂ detector gas reveals a different pattern of chromatographic peaks which remain to be identified. TID-1(TID-10)-N₂ detection is known to respond to heteroatom compounds like Oxygenates, Thiols, and compounds containing the Pyrrole functional group.

Like the B20 sample, CCID (TID-1-O₂) selectively detects the Linear Alkanes and the main FAMES, Palmitic Acid, Oleic Acid, and Stearic Acid Methyl Ester.

Petroleum standard solutions are often provided as dilutions in Methylene Chloride or Carbon Disulfide because an FID has reduced responses to these solvents. However, CCID and TID detection are very responsive to these solvents. Therefore, to avoid a large solvent upset, the heating current to the ceramic ion source was turned OFF during solvent elution, and returned to its normal operating value afterward. This turn OFF/ON of the ion source heating current was accomplished automatically as a run time event using the NPD electronics on a Thermo GC.

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



CCID chromatograms for two different Biodiesel samples - B100 Soy-Based (NIST SRM 27720) and B100 Animal-Based (NIST SRM 2773). Both samples were injected neat. The main FAME constituents - Palmitic, Oleic, and Stearic - were off-scale at the displayed signal amplification. Data obtained with DET retrofit detector hardware mounted on a Thermo Trace Ultra GC and powered with Thermo's NPD electronics. Oxygen was supplied through the 3 detector gas lines.

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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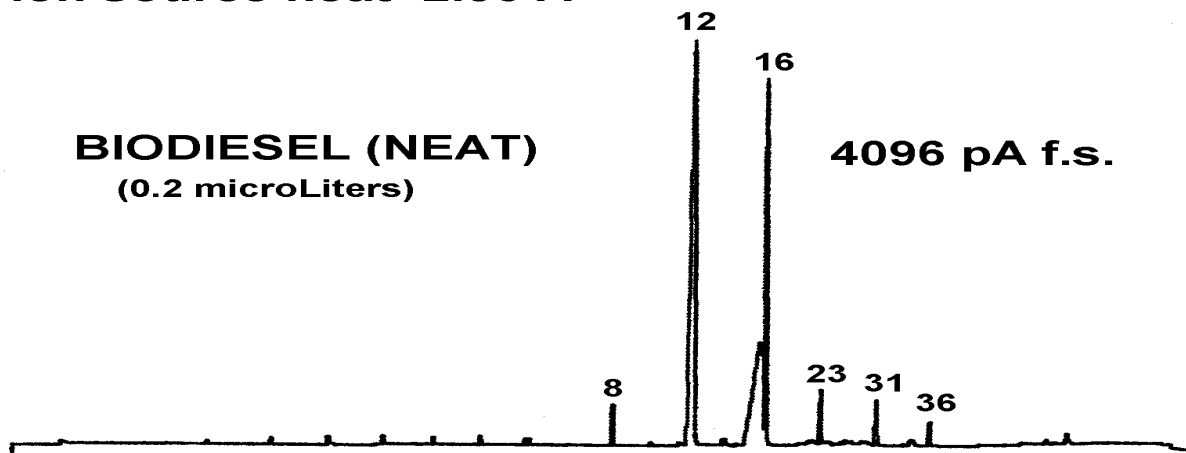
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**TID-1-Air (air=50, nitr.=5)
ion source heat=2.30 A**

Agilent 6890 GC

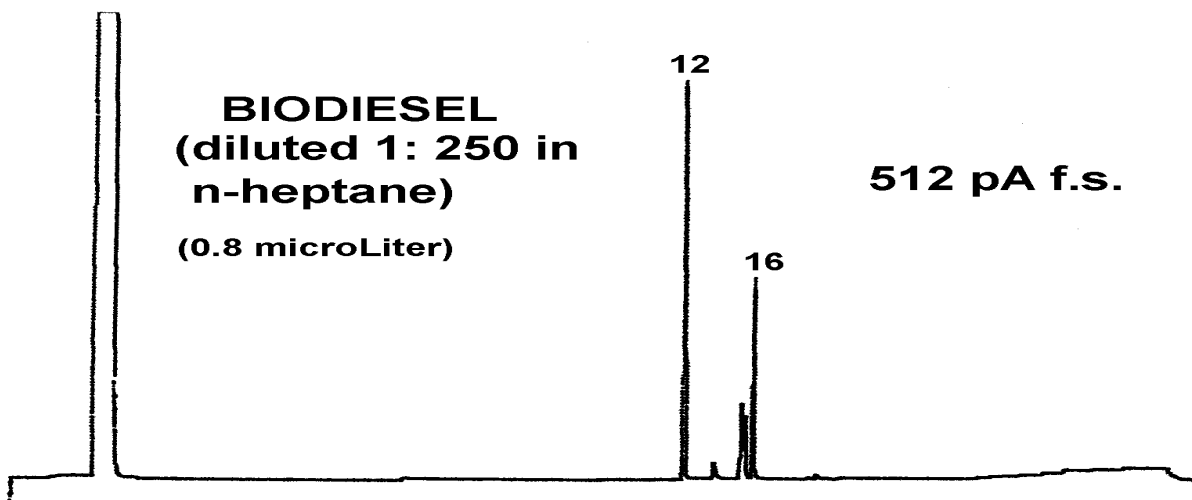
**BIODIESEL (NEAT)
(0.2 microLiters)**

4096 pA f.s.



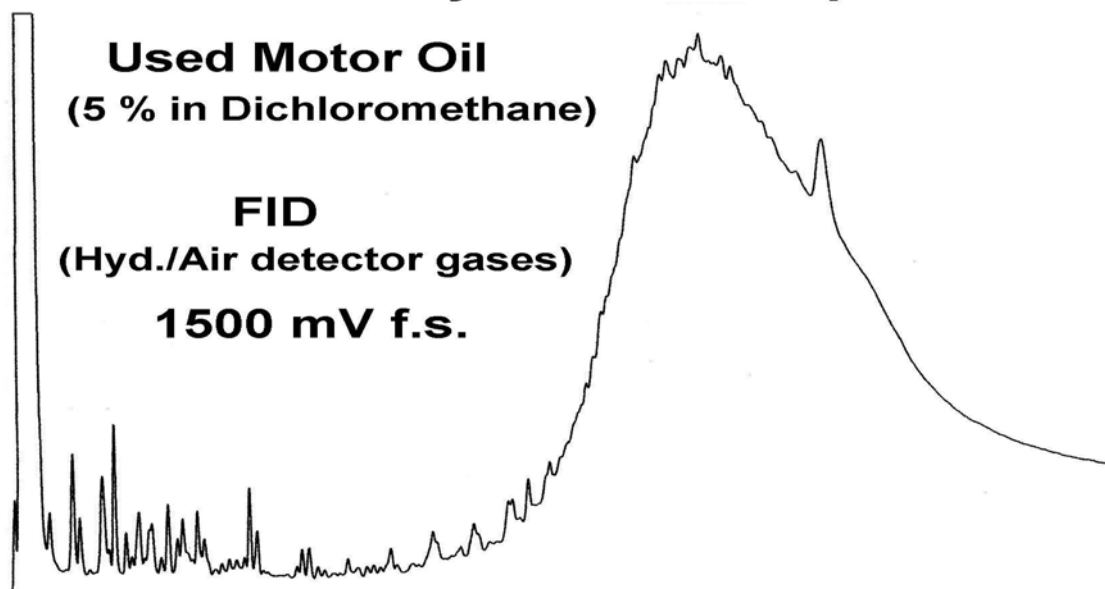
**BIODIESEL
(diluted 1: 250 in
n-heptane)
(0.8 microLiter)**

512 pA f.s.



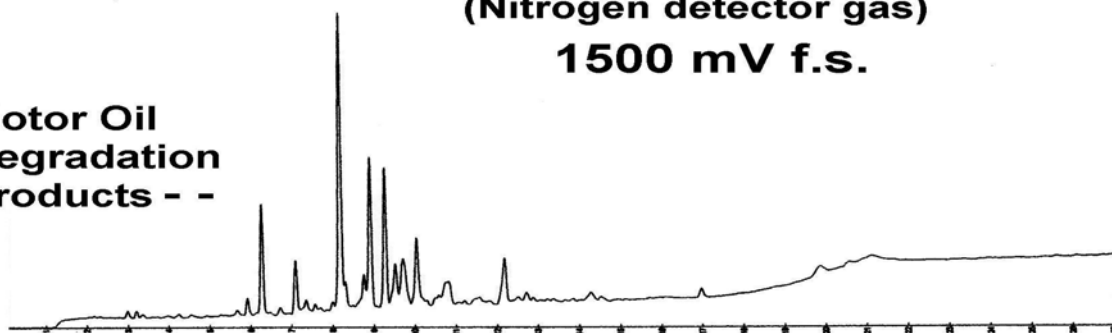
Agilent 6890 NPD Equipment with DET TID-1 Ion Source and DET Current Supply. All Labeled Peaks are Saturated FAME Compounds. Biodiesel Fuel Sample was made from Recycled Restaurant Frying Oils. Peaks 12 and 16 are Palmitic and Stearic FAMES, respectively. Peaks 8, 23, 31, and 36 are other saturated FAMES. Dilution of sample can remove some interfering peaks as they are reduced to concentration levels below that required for Catalytic Combustion ignition.

Motor Oil Analysis on a Compact GC

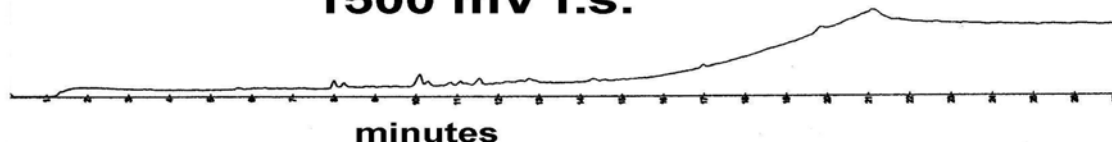


Used Oil: TID-1 Detector
(Nitrogen detector gas)
1500 mV f.s.

Motor Oil
Degradation
Products - -



New Oil: TID-1 Detector
1500 mV f.s.

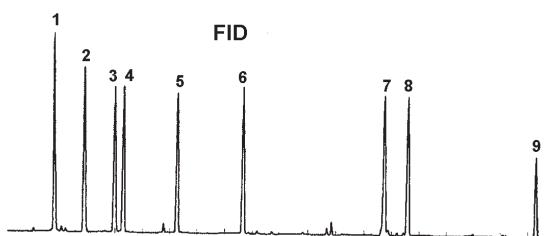


Selective TID-1 ionization in a Nitrogen detector gas environment reveals motor oil degradation products that build up with time as the oil is used. TID-1 monitoring of car oil vs. use can be very simple - take oil drop from car dipstick, dilute 1:3 in n-Hexane, and inject into GC. These data were obtained with interchangeable FID and TID modes on a compact SRI 310 GC equipped with DET detector hardware and a DET stand-alone Current Supply.

BRANCHED ALKANES Catalytic Pyrolysis Ionization (Nitrogen) & Catalytic Combustion Ionization (Oxygen)

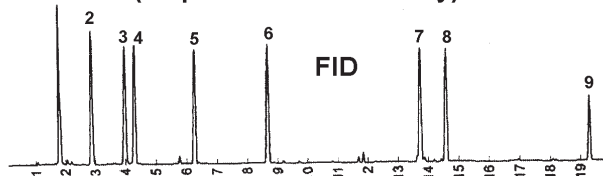
BRANCHED ALKANES SAMPLE

approx. equal concentrations of 1 - 8, lower conc. of 9



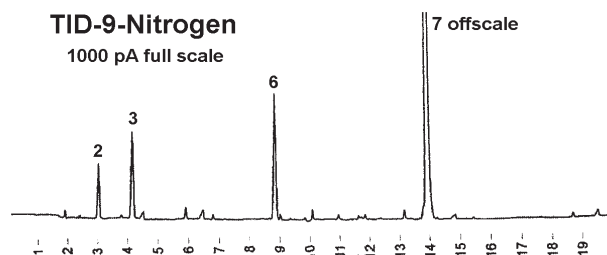
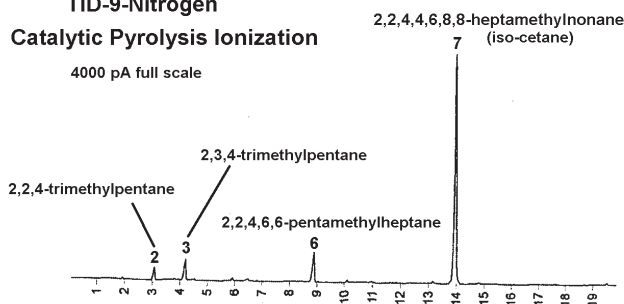
BRANCHED ALKANES SAMPLE

(amplified TID sensitivity)



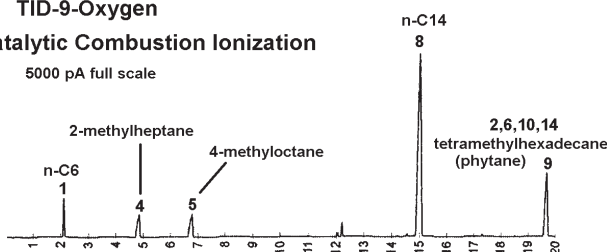
TID-9-Nitrogen Catalytic Pyrolysis Ionization

4000 pA full scale

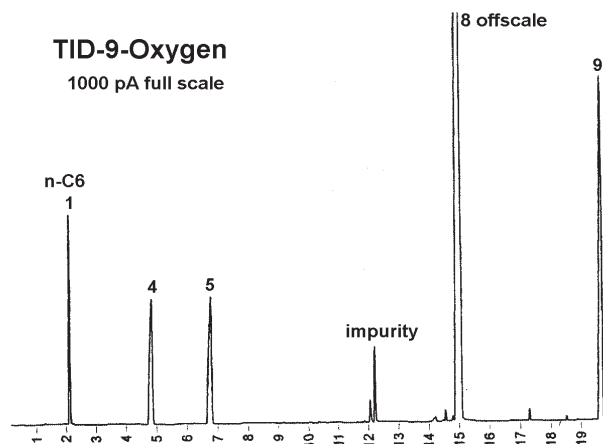


TID-9-Oxygen Catalytic Combustion Ionization

5000 pA full scale



TID-9-Oxygen 1000 pA full scale

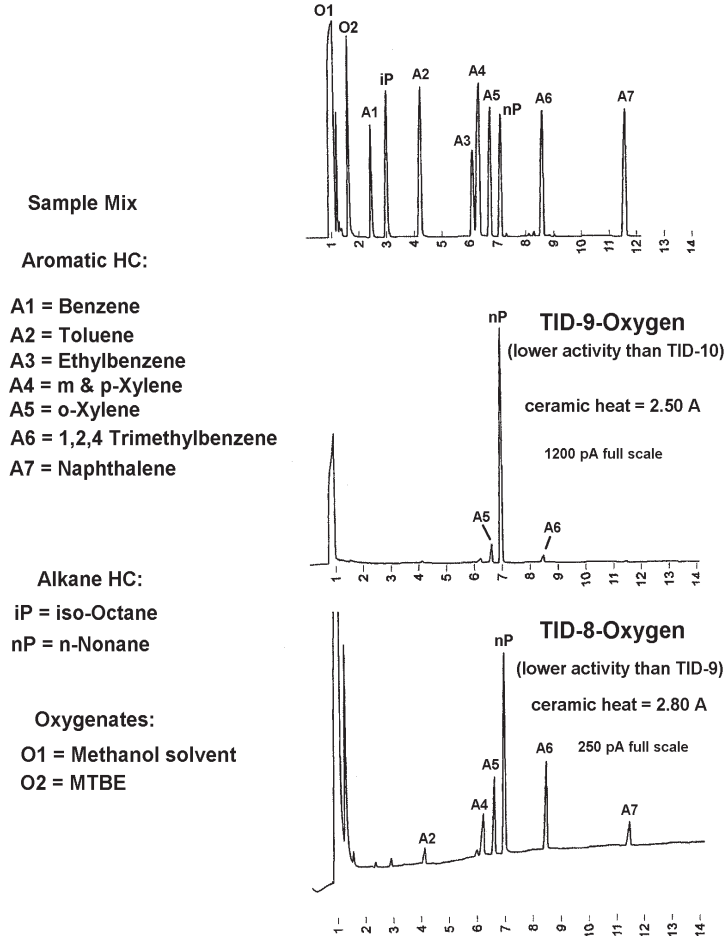


Many illustrations of selective CCID detection are accompanied by a comparison chromatogram generated using the same equipment with a Nitrogen detector gas environment rather than Oxygen. In the absence of Oxygen, selective ionization of high concentration Hydrocarbons can be described as a Catalytic Pyrolysis Ionization Detection (CPID) process. These data illustrate differences between CPID and CCID responses depending on the number of branched CH₃ groups and whether a C atom has more than one branched CH₃ group attached to it. The TID-9 ceramic source used here has a slightly lower catalytic activity than a TID-10 source which means it required a little higher heating current to achieve a comparable response.

CCID DETECTION OF ortho-XYLENE

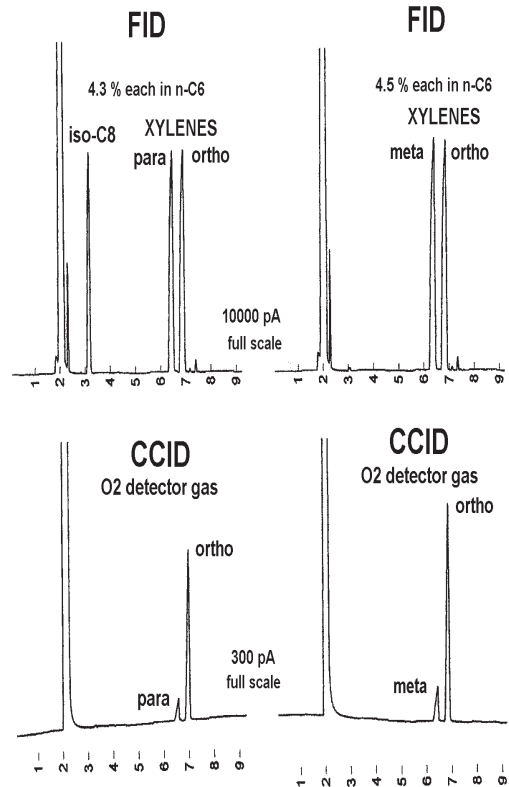
CCID DISCRIMINATION VS. AROMATICS

Comparison of Ceramic Ionizers with Different Catalytic Activity



CCID SELECTIVE RESPONSE

ortho-XYLENE vs. meta, para-XYLENE

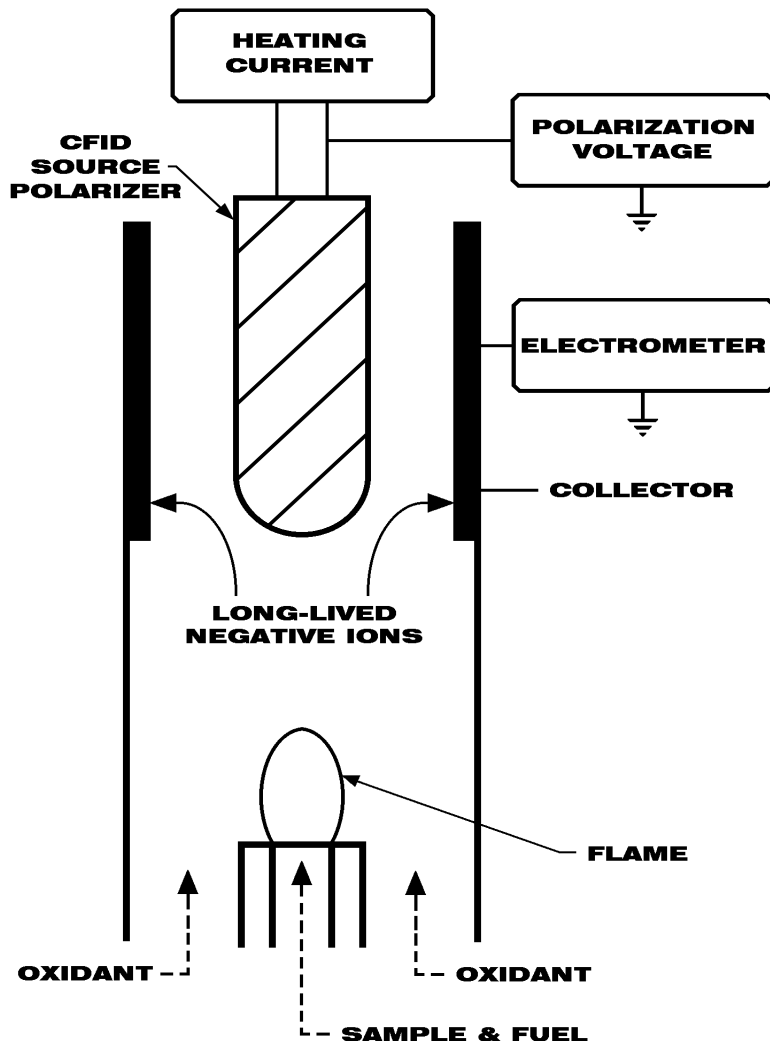


This sample has been analyzed previously with a TID-10 ion source to demonstrate CCID selectivity for Linear Alkanes vs. Aromatics. With a TID-9 ion source having a lower catalytic activity than TID-10, there was evident some small response to o-Xylene and 1,2,4-Trimethylbenzene, although the linear alkane n-Nonane response was still dominant. By substituting a TID-8 ion source with even lower catalytic activity, and operating it hotter, response to other Aromatics increased relative to the n-Nonane.

This comparison of CCID and FID responses illustrates that CCID with a high concentration of Oxygen as the detector gas environment, produced a much larger response from ortho-Xylene versus meta or para-Xylene. In other words, the Xylene isomer with CH₃ groups located in two adjacent positions on the Benzene ring is more easily ignited in combustion than isomers with CH₃ groups positioned further away from each other.

REMOTE FID

Selective Detection for Compounds Containing Pb, Sn, Si, or P Atoms



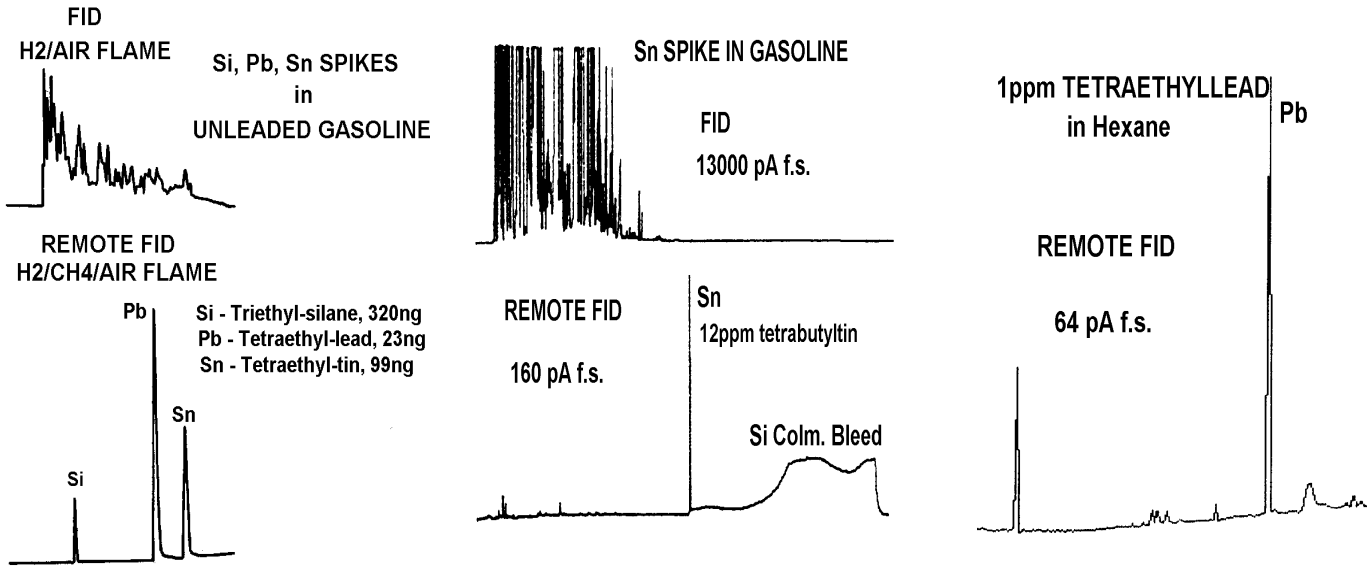
Signals from a conventional FID detector are due to gas phase processes that generate both positive and negative ions, and ion currents are sensed by having an appropriate polarization voltage move either one or the other polarity ion species to a collector electrode located near the flame jet. When polarizer and collector electrodes are instead positioned several centimeters downstream of the flame jet, the gas phase positive and negative ions produced from Hydrocarbon combustion recombine, and do not contribute to ion current at the collector. However, compounds containing Pb, Sn, Si, or P atoms form stable negative ion species in combustion. These stable ions persist to be measured at the collector, thereby providing selective detection for these heteroatoms. Selectivity versus Hydrocarbons can be enhanced by using a Hydrogen/Methane mix for the flame fuel rather than just Hydrogen. For P detection, an organically-fueled Remote FID is not as sensitive as an NPD, but it has a more uniform response per P atom, better selectivity vs. Hydrocarbons, and does not respond to N compounds.

DET

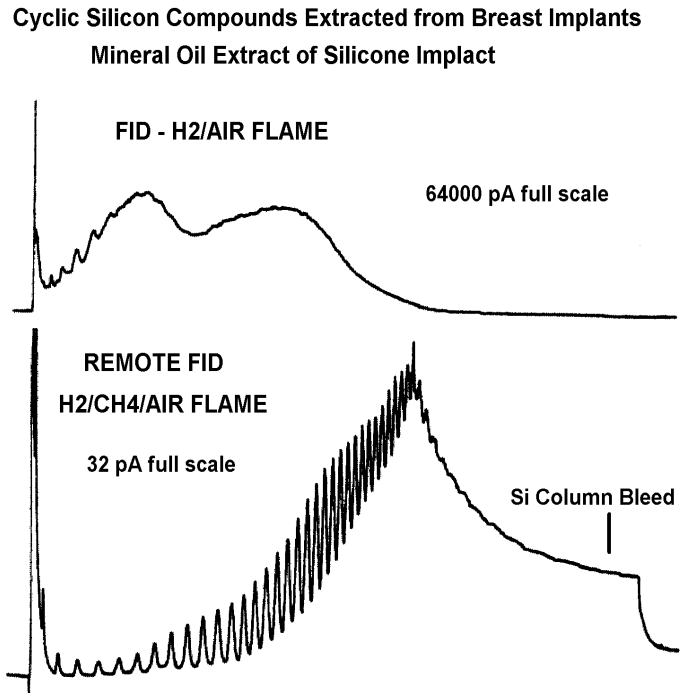
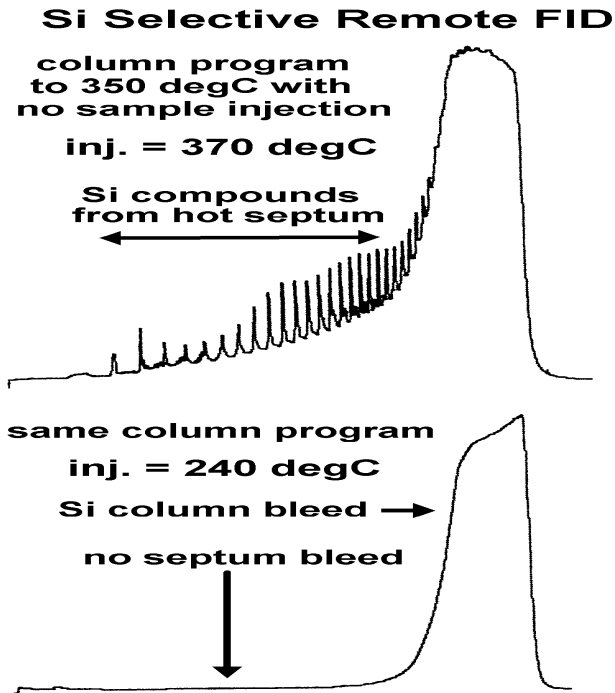
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REMOTE FID SELECTIVE DETECTION OF Si, Sn, Pb COMPOUNDS



Some data were generated with a DET Remote FID tower fitted onto an HP5890 FID/NPD detector base, and some data were from a DET Remote FID tower fitted to a Varian 3700/3800 FID/TSD base. CFID ceramic ion source installed in tower structure was heated to ignite the flame, then functioned as the polarizer electrode. Flame fuel consisted H₂ and 20%CH₄. Ion source was powered by a DET Current Supply. Signals on the Varian GC were measured with an existing Varian TSD electrometer. HP5890 signal measurement required a stand alone electrometer from Keithley. HP5890 hardware also fits FID base on Agilent 6890.



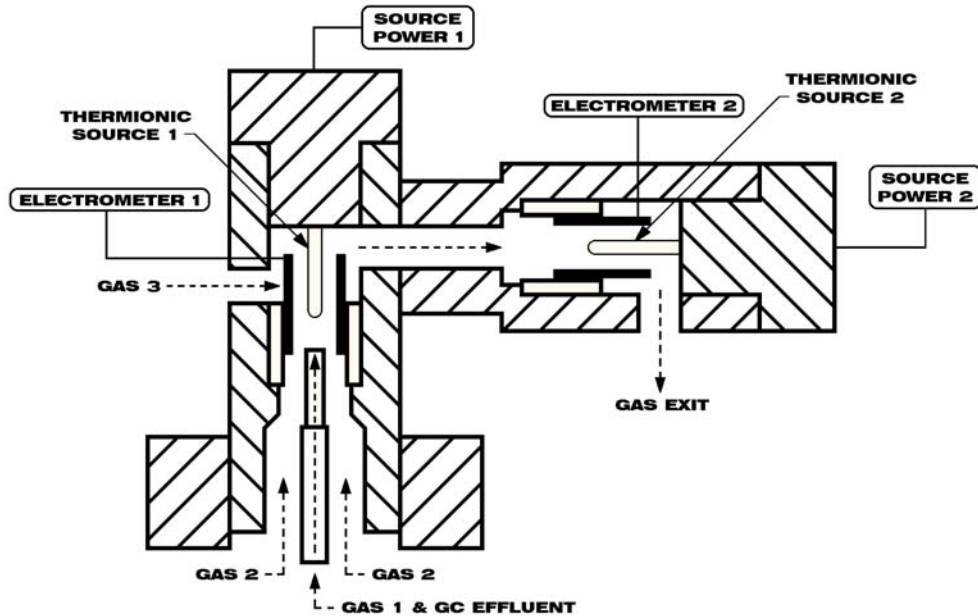
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TANDEM TID

2 simultaneous signals per sample with many possible combinations providing the ultimate in selective detection possibilities



Detector hardware is designed to custom retrofit onto an FID/NPD detector base already existing on a GC - detector gases 1 and 2 of various compositions are supplied through the 2 gas lines normally used to supply H₂ and Air to the base - detector gas 3 is used to purge the volume between the 2 detection stages - separate ion sources and supporting electronics are used for each stage. Tandem detector hardware is available to fit Thermo, Varian/Bruker, SRI Instruments, and HP 5890 GC models. Thermo NPD electronics are optimum for operating all modes, while Varian/Bruker TSD and SRI NPD ion source power supplies limit the response achieved for modes other than NPD. A stand-alone DET Current Supply corrects this deficiency of the Varian/Bruker and SRI electronics. NPD electrometers on Thermo, Varian/Bruker, and SRI GC models all suffice for signal measurement. HP 5890 retrofit equipment requires both a stand-alone ion source power supply and a stand-alone electrometer for signal measurement.

Signal combinations could include first stage detection that is non-destructive (e.g., TID-1), and destructive detection in the second stage (e.g., NPD, FID); or destructive first stage detection and second stage detection of first stage decomposition products; or two non-destructive stages with different ion sources and/or different gas environments; or the first stage could be used to catalytically convert non-detectable sample compounds into products that are detectable by the second stage of detection.

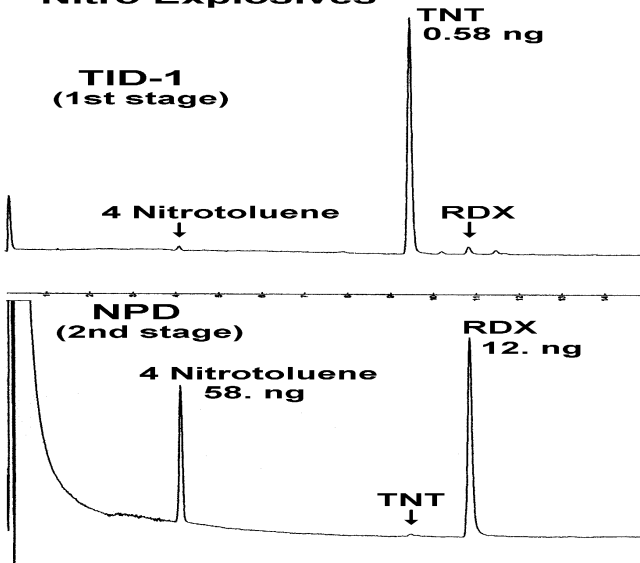
NO OTHER DETECTOR TECHNOLOGY PROVIDES SO MANY OPTIONS

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DETECTOR Engineering & Technology, inc.
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TANDEM TID on SRI 8610 GC 1 sample, 2 simultaneous signals Nitro Explosives

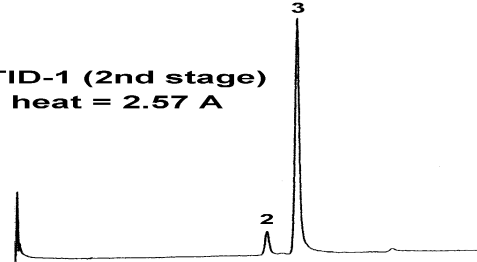


Series combination of DET TID-1 and NPD hardware mounted on SRI 8610 GC provided 2 simultaneous signals. TID-1 provided exceptional sensitivity to TNT while NPD responded in relative magnitude to all Nitro compounds.

NPD (1st stage) heat = 3.30 A

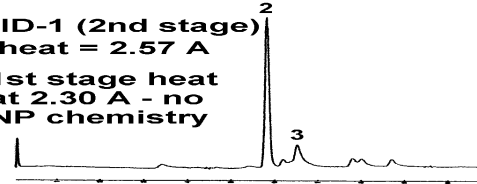


TID-1 (2nd stage) heat = 2.57 A



TID-1 (2nd stage) heat = 2.57 A

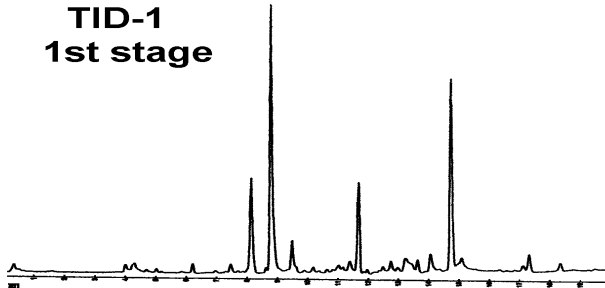
1st stage heat at 2.30 A - no NP chemistry



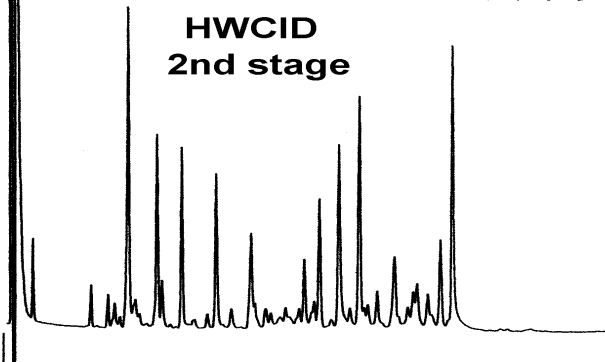
Simultaneous NPD/TID-1 of pesticides with destructive NPD in 1st stage. Bottom chromatogram shows change in TID-1 selectivity with 1st stage chemistry turned off.

Poison Eau de Cologne

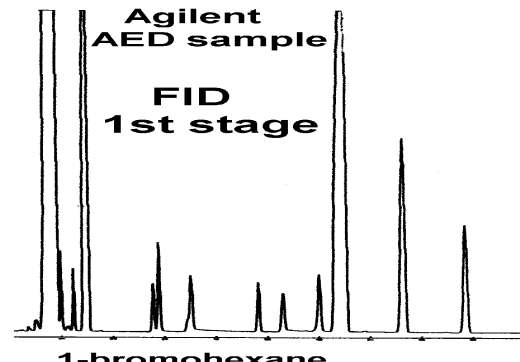
TID-1 1st stage



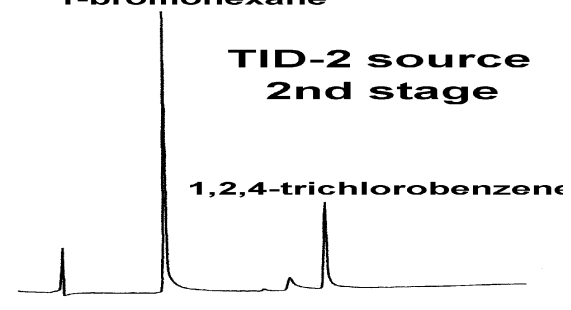
HWCID 2nd stage



Tandem combination of selective TID-1 and universal HWCID detectors. HWCID (Hot Wire Combustion Ionization) maintains a flame like environment adjacent to a continuously heated bare wire.

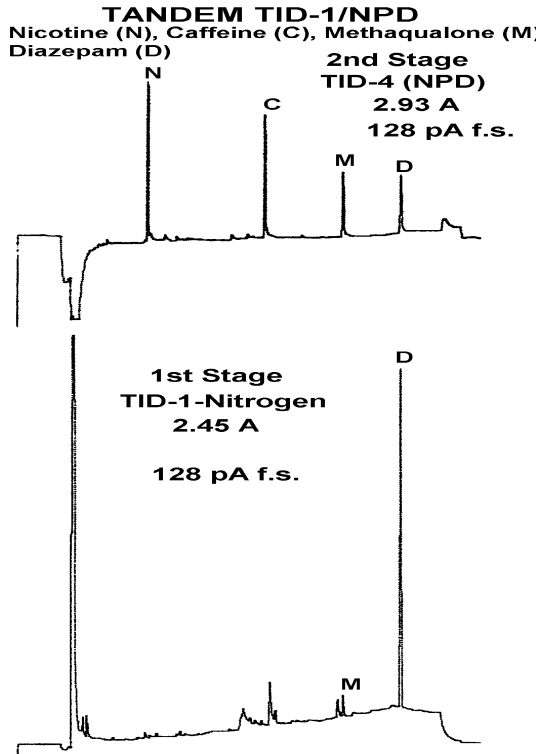


TID-2 source 2nd stage

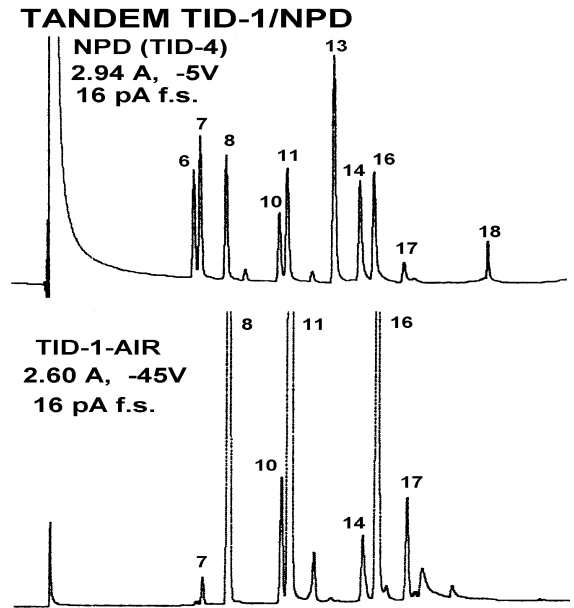


Simultaneous FID and FTID-2 detection where TID-2 ion source selectively detected Halogenated combustion products in effluent from 1st stage flame. Agilent AED sample contained a mix of high concentration Alkanes plus lower concentrations of N, Cl, Br, S, O, and Si heteroatom compounds.

EXAMPLES OF TANDEM SIGNAL COMBINATIONS

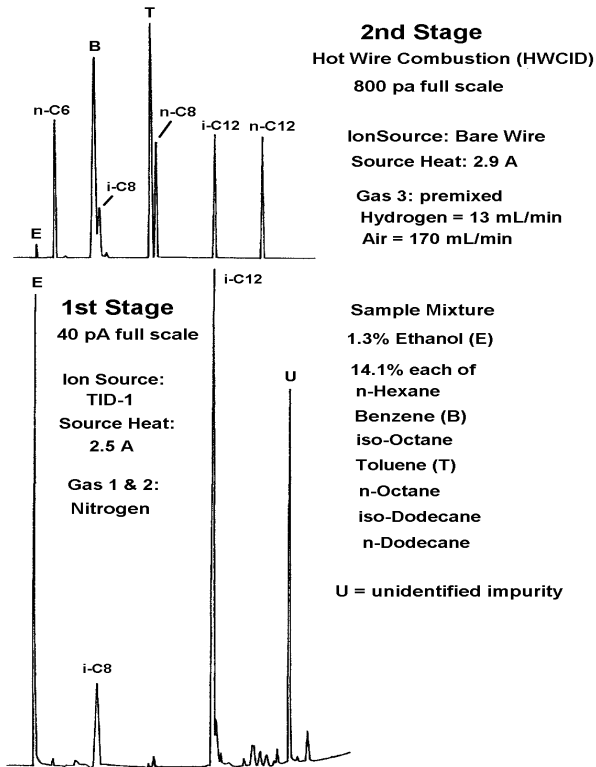


Drug mix. Tandem TID/NPD on Varian 3800 GC.

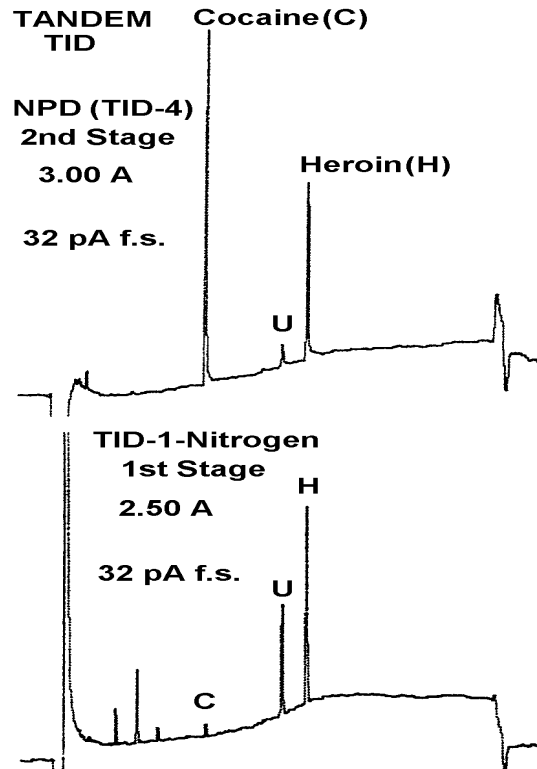


Tandem TID/NPD on HP 5890 GC.
 Explosives sample, 5 ng each compound:
 6=1,3-dinitrobenzene; 7=2,6-dinitrotoluene;
 8=2,4-dinitrotoluene; 10=1,3,5-trinitrobenzene;
 11=TNT; 13=RDX; 14=4-amino-2,6-dinitrotoluene;
 16=2-amino-4,6-dinitrotoluene; 17=tetryl; 18=HMX

TANDEM TID/HWCID



HWCID - universal flame around hot wire.
 TID-1 - selective to Ethanol and Branched Alkanes



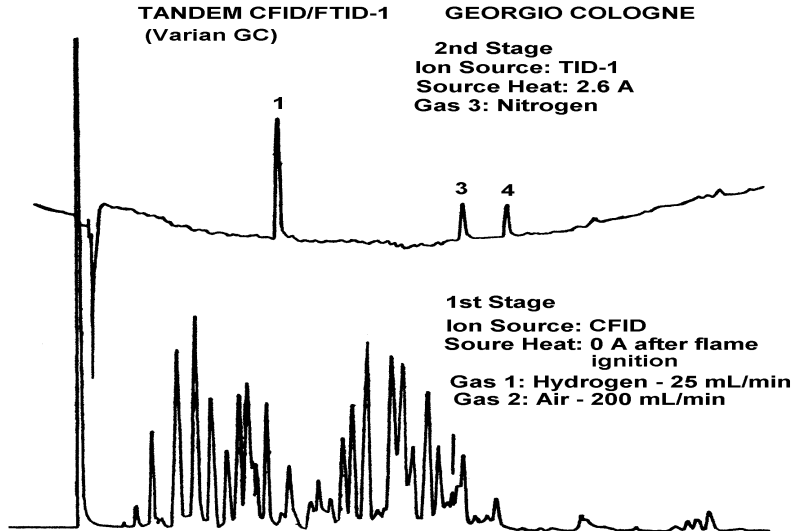
Tandem TID/NPD. Mix of Cocaine & Heroin standards. Varian 3800 GC. U = impurity in Heroin.

DET

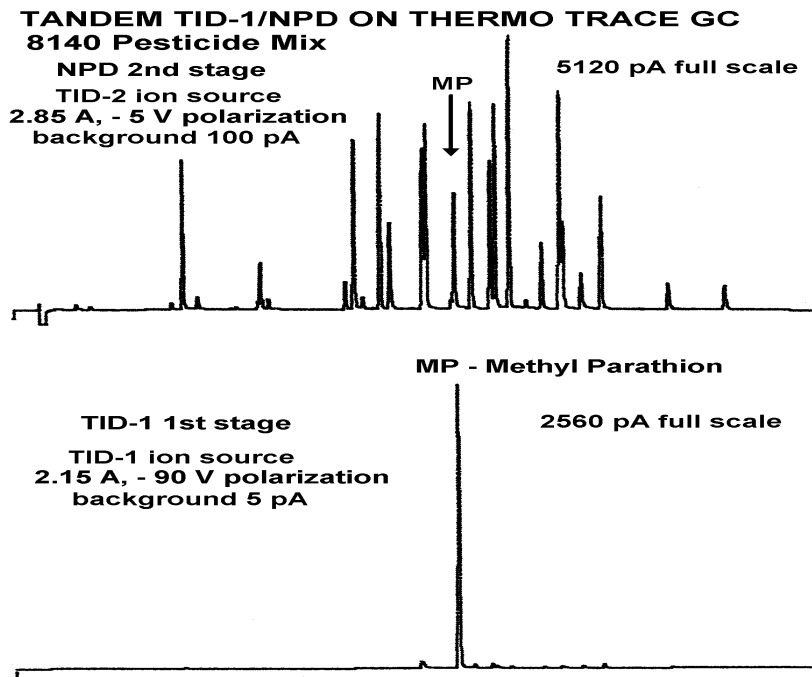
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Examples of Tandem Signal Combinations

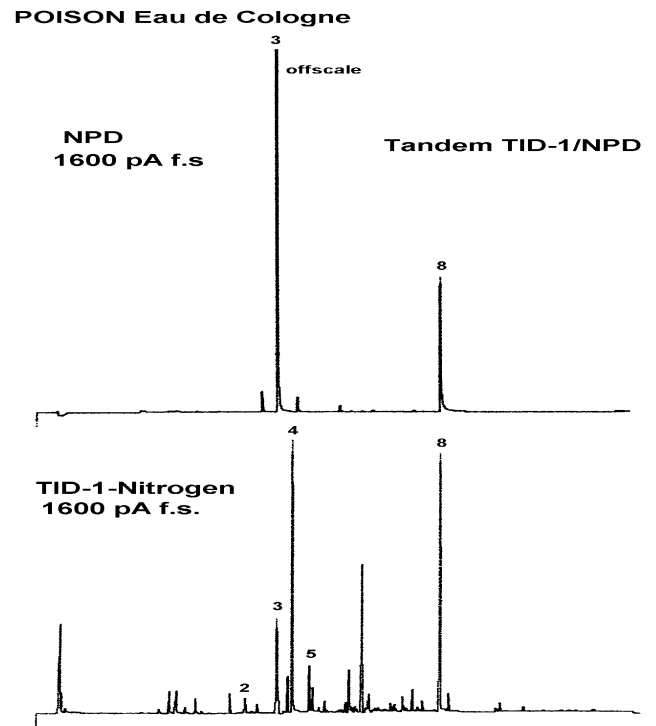
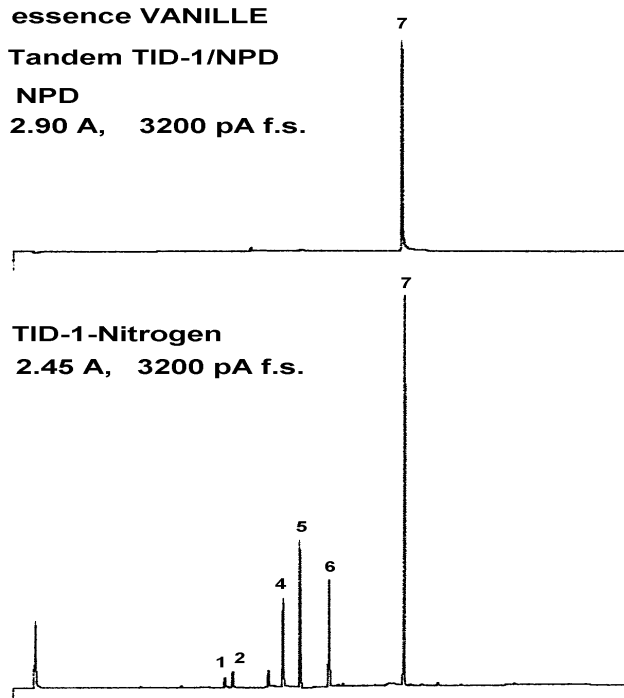
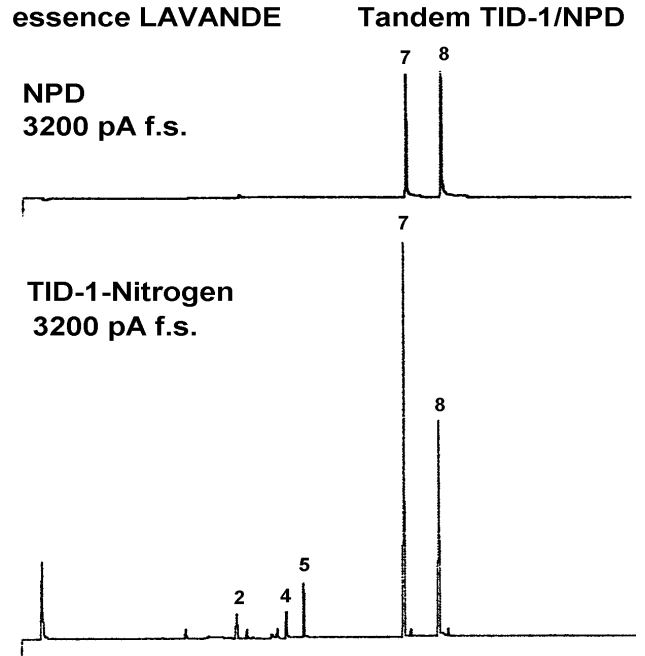
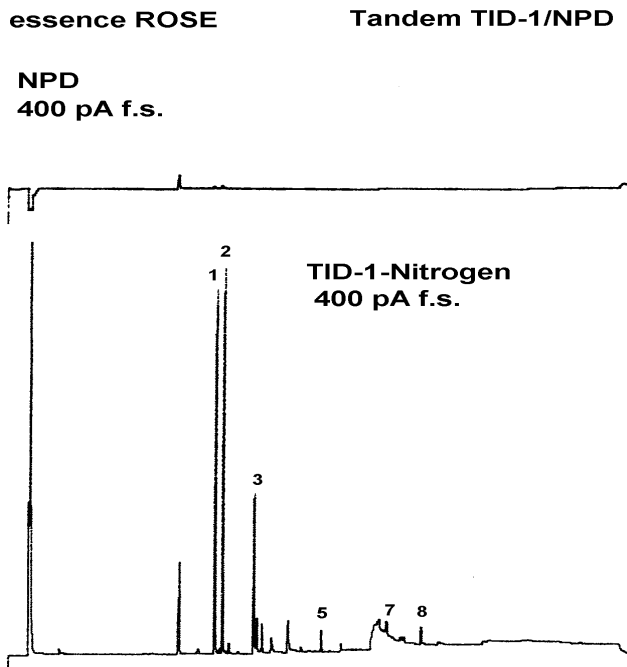


universal flame combustion in 1st stage, selective TID-1 detection of Nitrogen combustion products in 2nd stage.



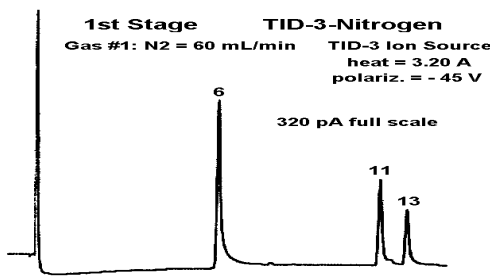
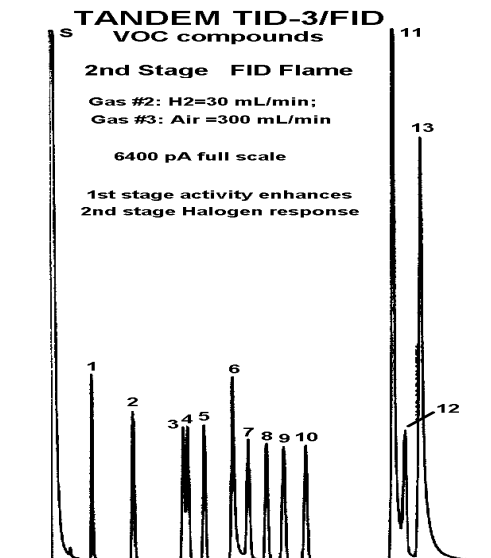
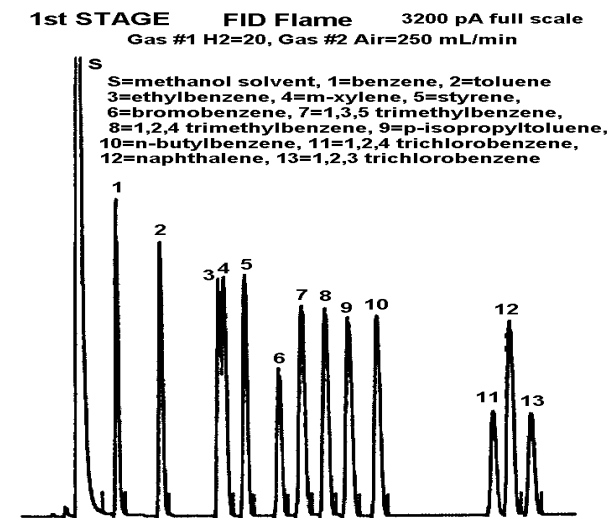
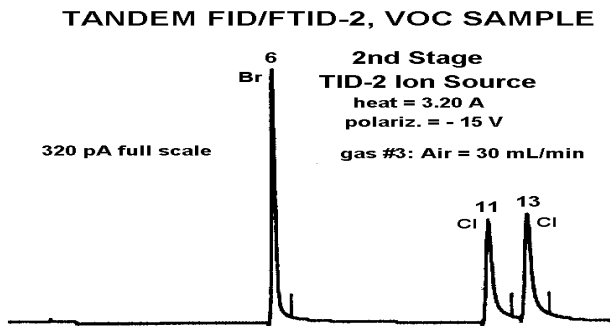
8140 mix of Organophosphorus Pesticides. First stage TID-1 selectively detects the NO₂ group in Methyl Parathion. Second stage NPD detects all the NP compounds. DET hardware & Thermo NPD electronics is an optimum combination for all NPD and TID detection modes.

TANDEM TID-1-NITROGEN/NPD - PURE FRAGRANCES & PERFUME

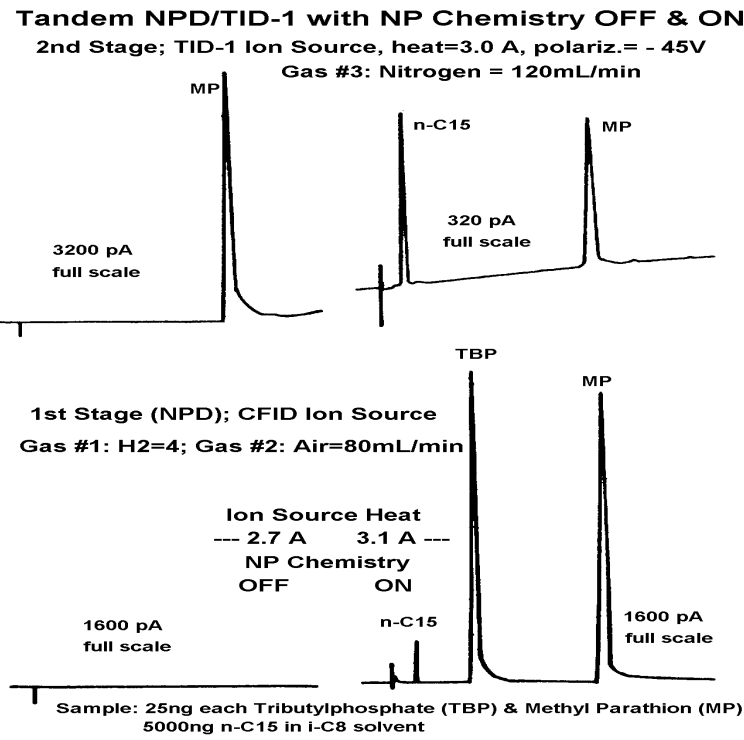


Simultaneous Signals - TID-1-Nitrogen 1st stage; NPD 2nd stage. Essence samples were 1% in Methanol. Same chromatographic conditions for all data. Peak labels refer to same retention times in all data. Varian 3800 with DET Retrofit Tandem hardware and DET Current Supplies for each detection stage.

TANDEM DETECTION - 1st STAGE CHEMISTRY CAN AFFECT 2nd STAGE



Same sample with FID in 1st stage vs. 2nd stage. 1st stage TID-3 activity enhances 2nd stage FID Halogenates.



2nd stage TID-1 response affected by whether 1st stage heating current is high enough to ignite NP chemistry.

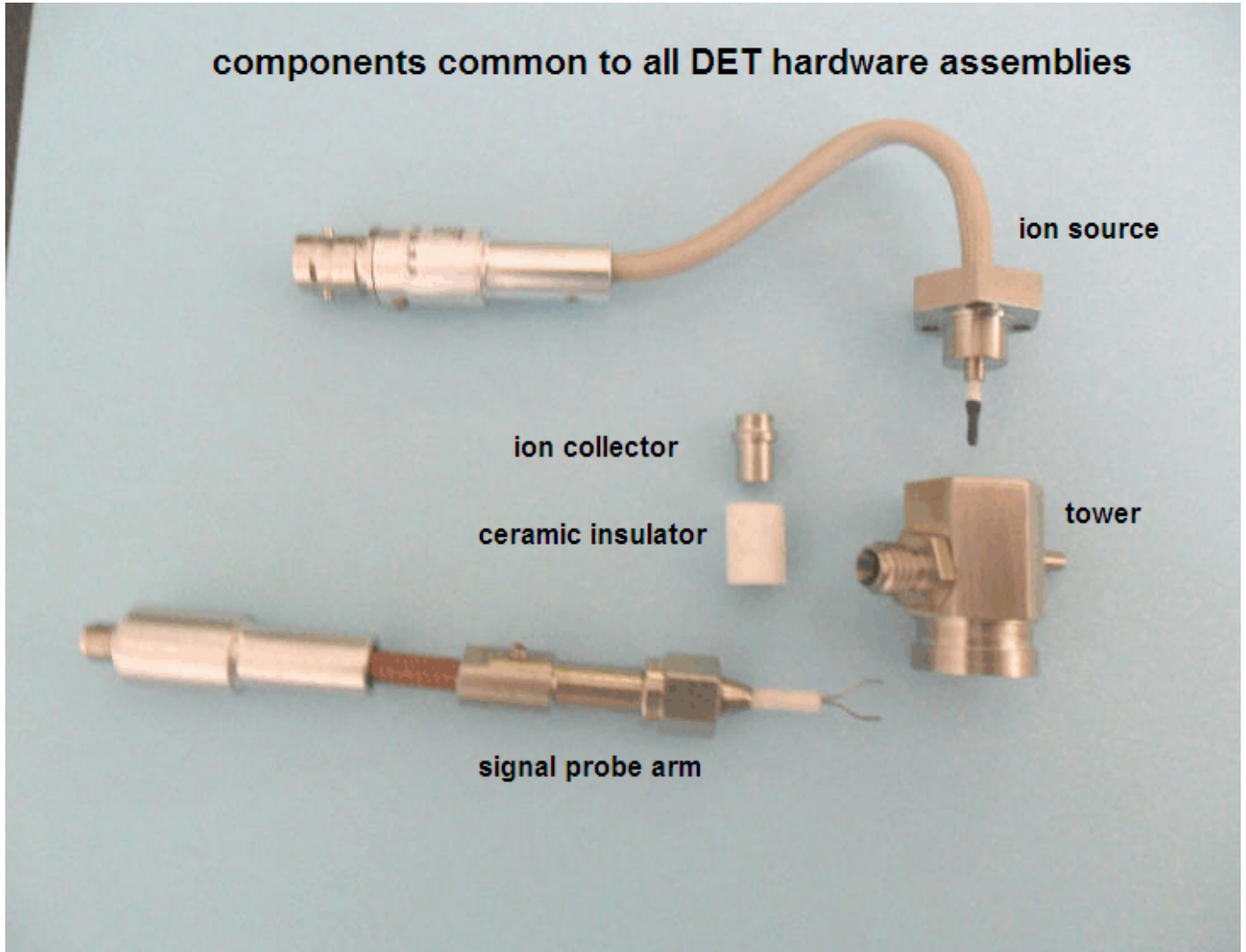
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DET Thermionic Detectors & Transducers

a simple design with only a few loose parts for easy, inexpensive maintenance



All DET structures have a common upper tower containing a collector cylinder and ceramic insulator, a signal probe arm, and the ion source. The tower base is custom designed to retrofit onto an FID/NPD base already existing on a GC, or it is provided with a choice of Swage or Tube inlet fitting for use as a stand-alone transducer. Samples and detector gases flow in through the bottom of the tower and out through the side. This structure configures the ion source and collector in a concentric cylinder geometry that provides streamlined gas flow and efficient ion collection within the detection volume. Top mounting of the ion source allows easy interchange of different ion source types for different modes of selective detection.

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STAND-ALONE DET CURRENT SUPPLY

Provides Constant Current heating and polarization voltage for powering DET's ceramic ion sources used in GC detectors, stand-alone transducers, or other applications requiring conversion of chemical compounds into measurable ion species (e.g., ion sources for mass spectrometers or ion mobility spectrometers). The electrical resistance of DET ion sources typically ranges from 0.7 Ohms when cold to 1.1 – 1.3 Ohms at operating temperature.



Thumbwheel set of ion source heating current (0 to 4.000 Amps) in 1 mA steps; Red/Green Status Light indicates open circuit or OK in ion source wiring – gives an immediate troubleshooting diagnosis in case of burnout of ion source wire core.



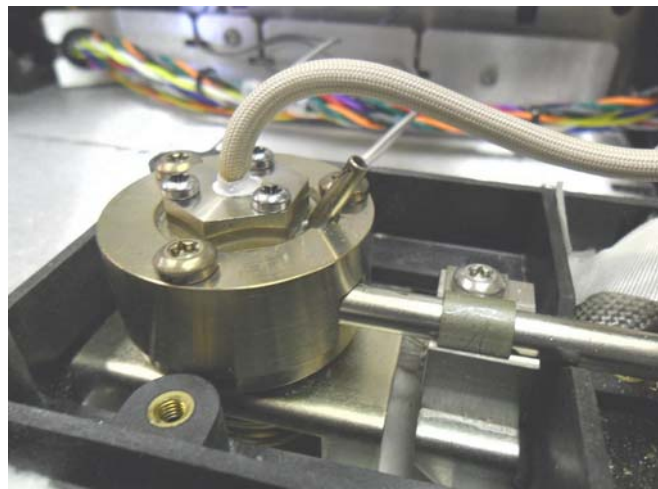
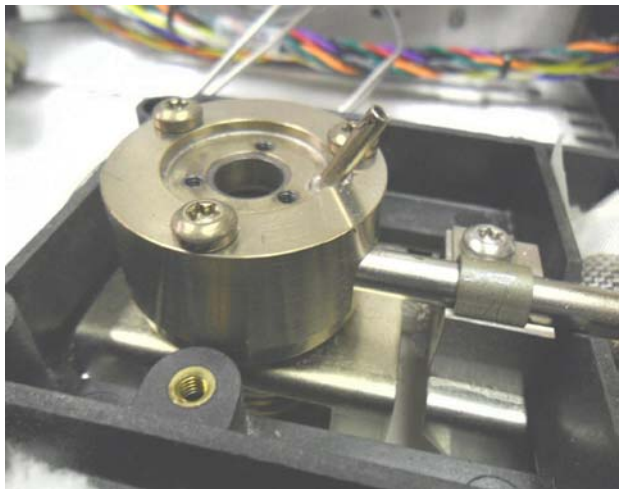
Switch selection of – 5, - 15, - 45 Volt ion source polarization (bias); – 5 V optimum for NP detection, higher polarizations provide 10 times better signal-to-noise for other detection. Twinex connectors on module & accompanying 4 foot long cable provide compatibility for powering ion sources on Agilent, Thermo, Varian/Bruker, and all DET NPD/TID equipment.
Dimensions: 5 ½ inches high, 6 inches wide, 10 ½ inches deep.

CERAMIC ION SOURCES FOR THE AGILENT 6890/7890 NPD



2 types of NP surface coatings are available for Agilent and all DET NPD retrofit equipment: TID-2 type (Black Ceramic) provides very sharp Phosphorus peaks for applications requiring P or both P and N detection (example, pesticides).

TID-4 type (White Ceramic) provides the best possible Nitrogen response for applications requiring only N detection (example, drugs).



The NPD detector structure on Agilent 6890/76890 GC models was designed to fit the hexagonal flanged mounting used on DET Ceramic Ion Sources. Therefore, any type Ion Source developed by DET can be used in the Agilent NPD hardware to extend the application of that equipment to other detection modes. Selective responses for Oxygenates, Halogenates, etc. is further enhanced by powering ion sources with a stand-alone DET Current Supply.

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DET PARTS FOR THE AGILENT TECHNOLOGIES 6890/7890 NPD Improved Performance - - Extended Detection Modes

THERMIONIC IONIZATION SOURCES (BEADS) - - - - \$495 ea

Durable cylindrical ceramic structure with a heater wire core, and mounted on a hexagonal shaped stainless steel flange. Different ceramic additives provide ion source types having different catalytic and ionizing activities. All DET sources are pre-tested for response before shipment, and are interchangeable in Agilent 6890/7890 NPD assemblies. Standard electrical connection is a Twinex type connector compatible with the Agilent NPD electronics, as well as a stand-alone DET Current Supply. Unlike the Blos glass NPD bead, DET ceramics do not soften at NP temperatures.

TID-10 (010-910-00) – selective response to **Oxygenates** (especially **Phenols, Carboxylic Acids, and Glycols**), **Nitro-compounds, some Halogenates, Pyrrole functional group**, and other electronegative functionalities - operates at 400 - 600°C in inert (N₂) or oxidizing (air, O₂) gas environments - femtogram detection for compounds like Methyl Parathion, 4-Nitrophenol, Pentachlorophenol, Heptachlor, TNT when powered with -45 Volt polarization available from a DET Current Supply

– TID-10 is also the key element used in the **Catalytic Combustion Ionization** mode which is selective for chains of **Methylene (CH₂) groups** in Petroleum Hydrocarbons, FAMES, or Triglycerides with no response to Aromatic or Cyclo-Hydrocarbons and with discrimination between compounds containing saturate vs. unsaturated Carbon bonds.

TID-2 (010-902-00) - NPD - selective response to **N,P compounds** - Black Ceramic coating has long life and minimal tailing of phosphorus compounds - operates at 600 - 800°C in a dilute H₂ in air gas environment - low picogram detection for NP compounds - lower cost alternative to Agilent NP sources.

TID-3 (010-903-00) - selective response to **Volatile Halogenates** - more uniform response to halogenates than TID-10 - operates at 600 - 800°C in inert (N₂) or oxidizing (air, O₂) gas environments - low picogram detection for Trihalomethanes with minimal peak tailing and greater response for Br versus Cl when powered with -45 Volt polarization from a DET Current Supply.

TID-4 (010-904-00) - NPD - our best coating for selective detection of **N compounds** (not recommended for P compounds because of tailing) - operates at same NPD conditions as TID-2 with 2 to 3 times better N detection.

TID-5 (010-905-00) - Halogen selective detection - more uniform response than TID-3 - operates at same temperature/gas environment conditions as TID-2, but with a higher polarization voltage (-45 V) available from a DET Current Supply - exceptional **selectivity for Br and I versus Cl** when configured with a stoichiometric H₂/air mixture.

TID-6 (010-906-00) - P selective detection with suppressed N response - uses much higher Hydrogen, Air, and Nitrogen flows than the TID-2 and TID-4 NPD modes.

TID-7 (010-907-00) - Green Ceramic for **Halogenated Pesticides, PCBs** - operates in N₂, Air, or O₂

FID Probe (020-902-00) – Uncoated wire used for **Universal Detection** with Hot Wire Combustion Ionization mode

DETECTOR CURRENT SUPPLY - - - - \$2244 ea

(001-901-01) for 115 V. Stand-alone module provides heating current and a selection of -5, -15, or -45 V polarization voltages for DET thermionic sources or the Agilent NP source. Recommended for use in place of the Bead Voltage supply on the Agilent NPD because it provides more stable Constant Current heating power for thermionic sources versus the Constant Voltage power provided by the Bead Voltage supply. Also, for modes of detection other than NP, the higher polarizations available from the DET supply provide as much as a factor of 10 improvement in signal-to-noise versus the fixed low polarization available from the Agilent supply. The DET supply also includes a green/red status light to immediately indicate that the source has burned out or the source power cable is not connected.

WIDE BORE JET & COLUMN SPACING KIT

Allows capillary columns of 0.53 mm diameter or less to be inserted through the jet to a termination close to the ion source as defined by a spacer tool. Eliminates sample degradation from interaction with jet metal; eliminates jet clogging from sample matrices; and eliminates the need to ever replace the jet.

(010-886-13) - column spacer & 64 mm long jet for Agilent's adaptable fitting NPD base - - - \$190 ea

(010-887-13) – column spacer & 43 mm long jet for Agilent's dedicated capillary base - - - - \$250 ea

DET innovations in chemical detection



DET RETROFIT NPD/TID/FID TOWER MOUNTED ON THE THERMO SCIENTIFIC TRACE GC. DET tower provides an optimum concentric cylinder geometry for stream-lined gas flow and efficient ion collection, and accommodates the same hex flanged ion sources as AGILENT GC models



CONTRAST BETWEEN THERMO'S NPD STRUCTURE AND DET'S NPD/TID/FID.

THERMO NPD (top) uses a DET ion source in the 1/4 inch tube mounting configuration and inserts the ion source into the side of the tower (i.e., lower probe arm shown with a Teflon insulator).

The DET structure makes a better gas seal to the THERMO detector base, and the concentric cylinder positioning of the ion source relative to a collector electrode eliminates dead volume side cavities. Ion Sources mount into the top of the DET tower, and Ion Source power and signal measurement are compatible with THERMO'S electronics. The combination of DET hardware and THERMO NPD electronics provides the most versatile capability for changing from NPD to TID to FID detection by simple changes of the ionizing element and the detector gases.

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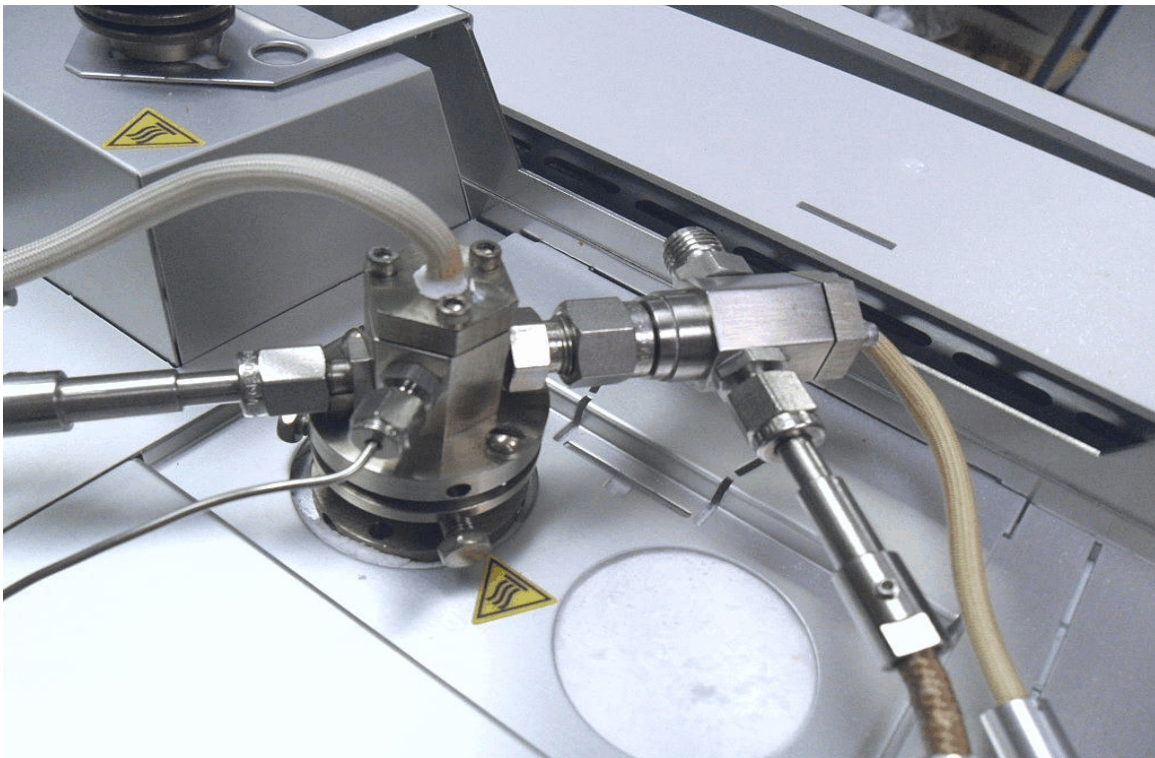
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TANDEM TID ON THERMO TRACE ULTRA GC

**2 simultaneous detector signals from 1 sample input
many different signal combinations are possible
TID/TID, TID/NPD, NPD/TID, FID/FTID, etc.**

**with multiple choices of DET's interchangeable ion source elements
TID-1, TID-2, TID-3, TID-4, TID-5, TID-6, TID-7, CFID, FID (bare wire)
and various mixtures of detector gases (N₂, Air, O₂, H₂)**



DET Tandem hardware fits onto Thermo NPD base - includes a ceramic tipped jet - 3 detector gases supplied through the "H₂", "Air", and makeup gas lines to the Thermo NPD base - 4th detector gas required to purge dead volume between the 2 detector stages - (1st stage can be purchased separately, 2nd added later).

THERMO NPD ELECTRONICS ARE CURRENTLY THE MOST VERSATILE AVAILABLE FOR POWERING ALL TYPES OF DET ION SOURCE ELEMENTS IN ALL MODES OF DETECTION - each detection stage needs a set of electronics.

(DET Tandem equipment is also available to retrofit Varian and SRI GC models)

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DET DETECTOR STRUCTURES FOR VARIAN/BRUKER GC MODELS (selective detection for N, P, O, Cl, Br, I, Pb, Sn, Si, NO₂, CH₂, Pyrrole, and other groups)



Varian/Bruker TSD on left, DET NPD/TID/FID in middle, DET Remote FID on right

NPD/TID/FID tower positions ion source near top of ceramic tip jet. Top mounting of ion sources provides an optimum concentric cylinder orientation of ceramic ion source and collector electrode, and allows easy access for ion source replacements. Interchangeable ion source elements provide multiple modes of selective detection with the same basic structure.

Remote FID tower positions ion source several centimeters downstream of jet. Remote FID detection mode burns samples in a flame at the jet, and selectively detects long-lived P, Pb, Sn, or Si ions that survive to reach the downstream collector. Remote FID tower can also be used for NPD and TID detection.



DET Tandem TID mounts on a Varian/Bruker FID/TSD detector base.

Tandem TID is a series combination of detector stages so that 2 simultaneous signals are obtained for each incoming sample. Many different signal combinations are possible through easy changes of the ion source element and detector gas environment in each stage.

DET ceramic coated ion sources can be powered with Varian/Bruker TSD electronics, although best response for Oxygenate and Halogenate selective modes is achieved with higher ion source polarization voltages as available from a stand-alone DET Current Supply.



DET NP/TID/FID Tower Assembly & Ceramic Tipped Jet Custom Fit onto Varian FID/TSD Detector Base.



Comparison of Varian TSD Tower/Bead (Top) & DET NP/TID/FID Tower/Ion Source (Bottom). Top Mounting of DET Ion Source Allows Easy Access for Ion Source Replacements.



DET Ion Sources are compatible with Varian's TSD electronics, and are the same standard configuration that fit Agilent 6890/7890 NPD equipment. There are 2 types of Ceramic Coating Choices for NP Detection:
Black Ceramic (TID-2 type) - formulated to give sharp Phosphorus peaks;
White Ceramic (TID-4 type) - formulated to give the largest possible Nitrogen response.

Other interchangeable Ion Source types include:
TID-1 type for Oxygenate selectivity;
TID-3 type for selectivity to Volatile Halogenates.

DET innovations in chemical detection



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.

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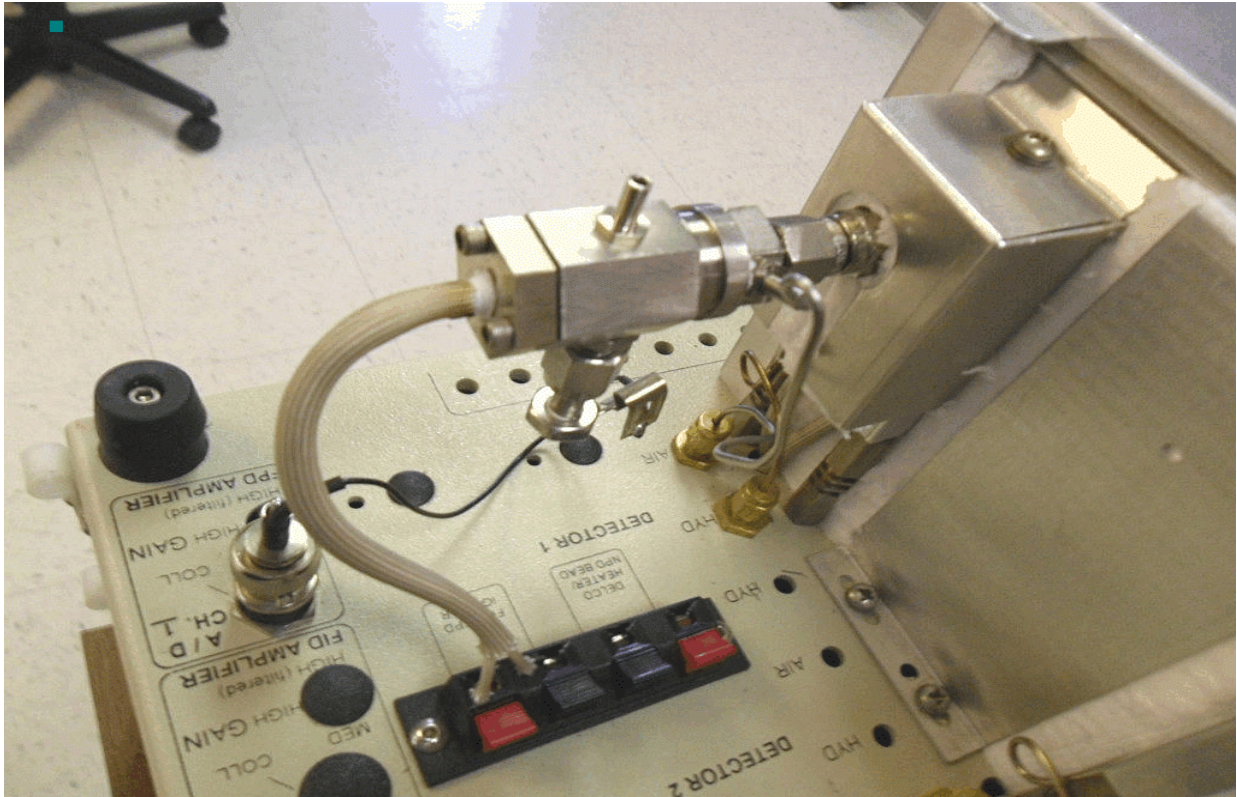
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DET NPD/TID/FID RETROFIT HARDWARE ON SRI GC ION SOURCE POWERED WITH SRI ELECTRONICS



DET retrofit hardware provides an optimum concentric cylinder configuration for ion source positioning relative to a collector electrode, and this contributes to streamlined gas flow through the detector volume and the most optimum electric field for ion collection. Ion sources have a universal mounting configuration similar to that used on Agilent and other GC models. The ion sources have bare wire terminations which are compatible with fitting into SRI's electronics connectors.

DET ion source elements can be powered by SRI's NPD power supply or also, with some care, by SRI's FID ignitor power supply. SRI electronics use a Constant Voltage method to heat the ion source rather than the preferred Constant Current method available with a stand-alone DET Current Supply. Voltage adjustments are set using screw driver settings, and in some GC models that involves raising the instrument top cover and disrupting the detector temperature controls. The SRI electronics are useable for either NPD, TID, or FID modes of detection. Compared to the stand-alone DET Current Supply, SRI electronics have less precise control of the ion source heating current, and are limited in ability to select the most optimum ion source polarization voltage for different modes of response.

DET REPORT

Stand-Alone Transducers

DETECTOR Engineering & Technology, inc.
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1.) FID, TID, AND NPD TRANSDUCERS FOR REAL TIME MONITORING OF VOLATILES IN AN AIR STREAM - SELECTIVE & UNIVERSAL DETECTION.

2.) REACTOR THERMIONIC IONIZATION ANALYSIS (RTIA) FOR SELECTIVE SCREENING OF NON-VOLATILE CONSTITUENTS IN LIQUIDS AND SOLIDS.

1.) FID, NPD, AND TID-1 TRANSDUCERS FOR REAL TIME MONITORING OF VOLATILE SAMPLES IN AN AIR STREAM.

Previous DET Reports have addressed the use of thermionic detectors like the NPD and TID-1 in stand-alone applications involving samples in an Air stream. Although these reports described detector hardware configurations that did not include a GC installation, they still included some means of providing external heating control of the detector body. However, DET detection transducers designed for stand-alone applications are small enough in physical size that some self-heating occurs from the detection mechanism operating within the transducer body. The present report describes using FID, NPD, and TID-1 transducers that had only a simple thermal wrap covering and no other external heat.

DET's experience of designing detectors to fit different brand GC instruments has led to a methodology for constructing detection structures with many different tube or Swage type inlet and outlet configurations. As a consequence, these detection structures or transducers can be easily mated to standard type gas line fittings. DET transducer bodies are constructed of stainless steel and ceramic materials so they are capable of withstanding operation up to 400°C.

Since Air is a primary detector gas supplied to thermionic detectors like the NPD or TID-1, as well as to an FID, it is a logical extension to consider using these detector types to sense samples carried in the Air stream rather than in an inert carrier gas stream eluting from a GC column. In the present work, the Air supply to the FID, NPD, or TID-1 transducers was simply ambient Air drawn into the transducer by attaching a sampling pump to the transducer's exit port. For gathering data, the transducers were simply supported on a laboratory ring stand, and connected to the appropriate stand-alone power and signal electronics.

FID Transducer for Samples in an Air Stream. In the conventional implementation of Flame Ionization Detection, samples are admixed with Hydrogen and enter

the flame through the center orifice of a jet structure, and Air is supplied around the outer periphery of the jet. In this manner, it is ensured that all the sample passes through the very core of the flame. With samples introduced with the Air supply around the outer periphery of the jet, it is clear that some sample molecules will likely bypass the actual flame. Hence, it is to be expected that an FID with sample compounds in the Air stream will not be quite as sensitive as the conventional FID with samples in the interior of the flame.

Hydrogen-Air flames are unique chemical environments characterized by high temperatures and high concentrations of radical or unstable chemical species such as H, O, and OH. Sample molecules are efficiently decomposed in such a reactive environment, and sample fragments are ionized in gas phase reactions with the flame radicals. For organic samples, an important mechanism is the chemi-ionization reaction $\text{CH} + \text{O} = \text{CHO}^+ + \text{e}^-$. This ionization occurs in that flame region where the CH and O radicals are at their highest concentrations, and that is the conical periphery of the flame where the Hydrogen and Air are mixing together. In a conventional FID design, a very small orifice jet is preferred in order to provide a tall thin flame where the ionization region is constricted as small as possible to the flame core. For an FID where samples are in the Air stream, a short broad flame is more desirable in order to have the ionization region extend out into as much of the Air stream as possible.

Figure 1 shows a schematic of the FID transducer used in this work. The jet was a ceramic tube of 0.125 inches O.D. x 0.062 inches I.D., and that was surrounded by an ion collector cylinder of 0.200 inches inner diameter. The collector electrode was insulated from the surrounding transducer body by a high purity alumina ceramic sleeve. A Bare Wire loop located downstream of the jet served the dual role as Flame Ignitor and Polarizer. The mounting flange for the Ignitor/Polarizer element was identical to

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Stand-Alone Transducers

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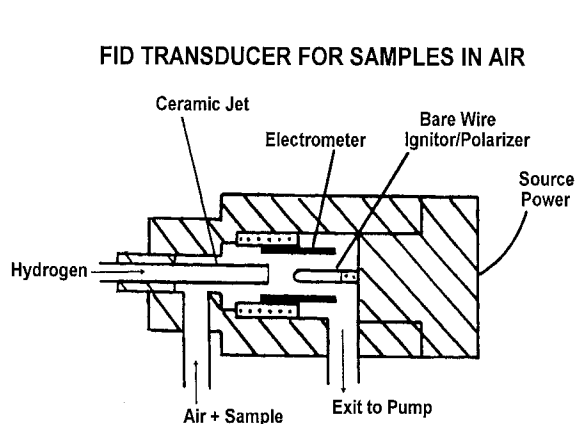


Figure 1. Schematic of FID transducer for Air samples.

that used on DET and Agilent 6890 NPD ion sources. Power for the Bare Wire FID Probe and signal measurement from the ion collector were provided by a DET Current Supply and Keithley 6485 Picoammeter.

The FID Transducer body incorporated a 1/8 inch Swagelok seal for the jet attachment, a 1/4 inch tube for the incoming Air and sample, and a 1/4 inch tube for the exit port. The jet was connected to a Hydrogen inlet line via a 1/8 to 1/16 inch Swagelok reducer, and the interior of the Air inlet tube had a glass wool packing to prevent detector spikes from incoming particulate matter. Hydrogen flow was provided by a DET Flow Control module which included an adjustable needle flow restrictor and on/off control without changing the flow setting. A calibration of flow versus gas supply pressure was used to determine the Hydrogen supplied to the FID as well as to an NPD transducer. For the FID data of this report, the Hydrogen flow was 30 mL/min.

In attaching a sampling pump to the exit of an FID transducer, consideration must be given to handling the potential condensation of Water vapor produced by the flame. Once the flame is ignited, the transducer body self heats in about 15 minutes so that condensation within the transducer no longer occurs. However, condensation can occur in the unheated tubing leading to the pump. To avoid this, we attached a gas tee configuration at the transducer exit, and used a 5 L/min pump to pull a 0.5 L/min Air sample through the transducer, and an additional 4.5 L/min Air purge through the exit tee. This purge gas eliminated condensation in the gas lines to the pump.

FID Transducer 400 pA/mV
 hydrogen = 30, air = 500 mL/min

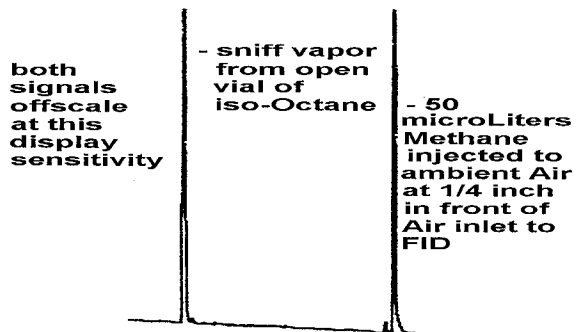


Figure 2.

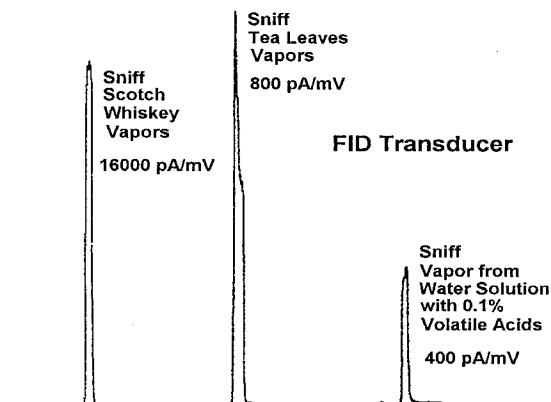


Figure 3.

Figures 2 - 5 illustrate data obtained with the FID Transducer. After first ignition of the flame, we allowed 45 minutes for thermal equilibration of the transducer and for all traces of moisture to be eliminated from inside the detector volume. Various types of samples were then brought to the vicinity of the transducer inlet to allow their organic vapors to be swept in with the ambient Air pulled into the transducer.

Figure 2 shows signals obtained from an open vial of iso-Octane, and from a small injection of Methane into the ambient Air located about 1/4 inch away from the transducer inlet. Figure 3 compares signals for vapors

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emanating from liquid samples of Scotch Whiskey (i.e., Ethanol response), and a 0.1% solution of Volatile Fatty Acids in Water. Also shown in Figure 11 is the signal obtained from vapors emanating from a solid sample consisting of the very aromatic leaves of a Herbal Tea (i.e., Good Earth brand). The large Tea Leaves signal was further compared in Figure 4 with signals from Ground Coffee and Coffee Beans. Figure 4 also shows signals generated from Human Breath samples exhaled and inhaled through a tube that terminated at an ambient Air exit location about 1/4 inch in front of the transducer inlet.

In order to ignite the FID flame initially, an electrical current was passed through the Bare Wire element to raise its temperature sufficiently for ignition. Once the flame was ignited, this heating current was reduced to zero, and the Bare Wire element then served as just a - 45 V polarizer to drive negative ions in the flame toward the collector electrode. However, once the transducer body heated up from the flame, we discovered that the flame autoignited with just the turn on of Hydrogen flow and no additional heating current applied to the Bare Wire. This effect is illustrated in Figure 5. The autoignition was attributed to catalytic processes occurring at the Bare Wire surface.

Also shown in Figure 5 is a comparison of signals from vapors evolved from a 1 gm Soil sample versus a similar Soil sample Spiked with 3 μ L of Diesel Fuel. For these data, the solid soil samples were distributed inside the bottom of a 50 mL open top vial and vapors at the top of the vial were sniffed by the FID Air inlet.

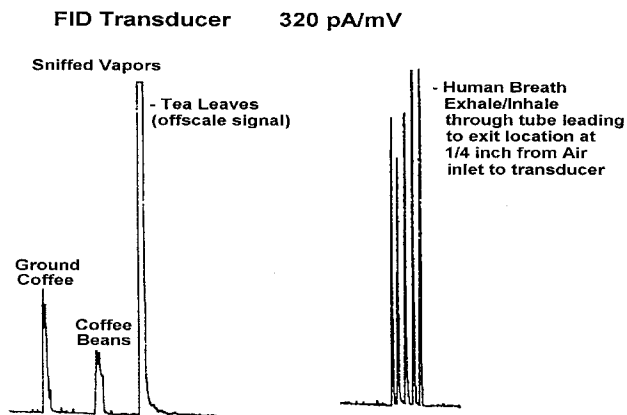


Figure 4

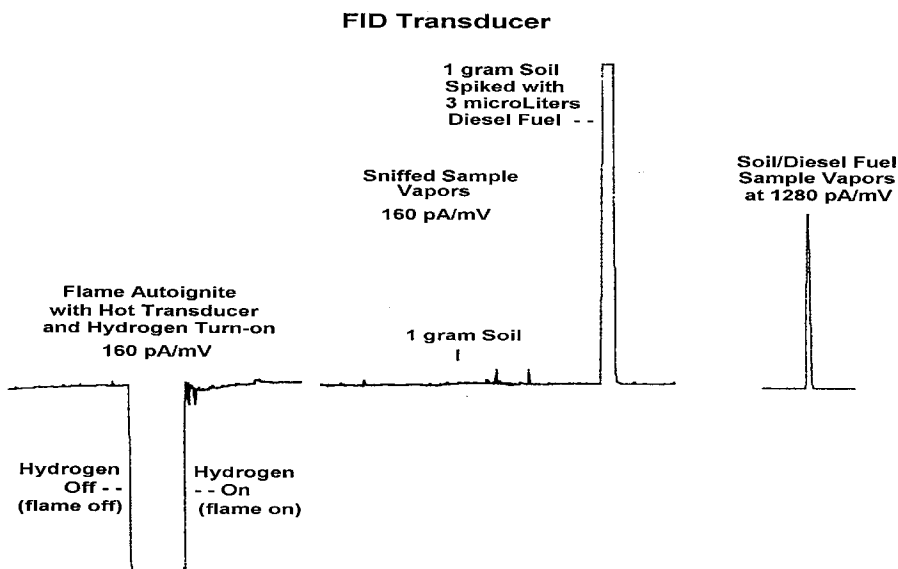


Figure 5. Demonstration of flame auto-ignition with just turn on of Hydrogen and no electrical heat applied to ignitor wire. Comparison of signals from soil samples with and without contamination by diesel fuel.

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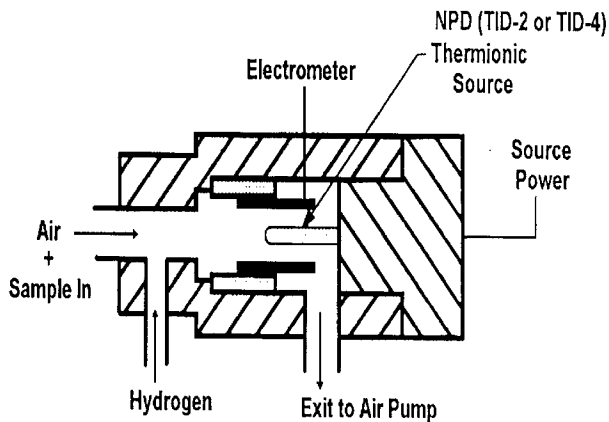
NPD Transducer for Samples in an Air Stream.

Like the FID, an NPD requires the addition of some Hydrogen to the incoming Air flow. However, there is no requirement for a jet structure, so the Hydrogen can simply be added with a gas tee at the transducer inlet. Figure 6 shows a schematic of the NPD Transducer used in this work. The thermionic ion source used in this work was a Black Ceramic TID-2 type. The main transducer body had a 3/8 inch O.D. tube inlet, and that was attached to a 3/8 inch Swagelok union and 3/8 to 1/4 inch Swagelok port reducer tube which provided the Air inlet path. The side of the 3/8 Swagelok union was modified with the addition of a tee connection providing a 1/16 inch Swagelok fitting for the Hydrogen inlet.

Like an FID, the Hydrogen - Air chemistry in an NPD also produces some Water vapor, but at a much lower concentration than the FID. Consequently, it was possible to connect the sampling pump directly to the 1/4 inch Swagelok outlet of the transducer without requiring any additional exit purge gas flow as was used with the FID. For the NPD data of this report, the Air flow was 350 mL/min and the Hydrogen flow was 9.8 mL/min.

Figure 7 shows some signals generated with the NPD Transducer. The Ground Coffee, Tea Leaves, and Diesel Soil samples were the same as in the FID data of Figures 4 and 5. In contrast to the FID response, the NPD exhibited a negligible signal for the Diesel Soil sample, and the signal for the aromatic Tea Leaves was small compared to the signal for the N-containing aroma compounds from the Ground Coffee. Meanwhile, a large signal was obtained from the vapors emanating from a Grain Fumigant that emits Phosphine when exposed to moisture. Figure 15 also exhibits a negative signal due to exposure to the vapors from an open bottle of iso-Octane. At high concentrations of Hydrocarbons in the incoming gas stream, the ignited chemistry in the NPD can be quenched as evidenced by a negative type signal.

NPD TRANSDUCER FOR SAMPLES IN AIR



NPD Transducer 12800 pA/mV
 Hydrogen = 9.8, Air = 350 mL/min
 Sniffed Vapors from Open Sample Vials

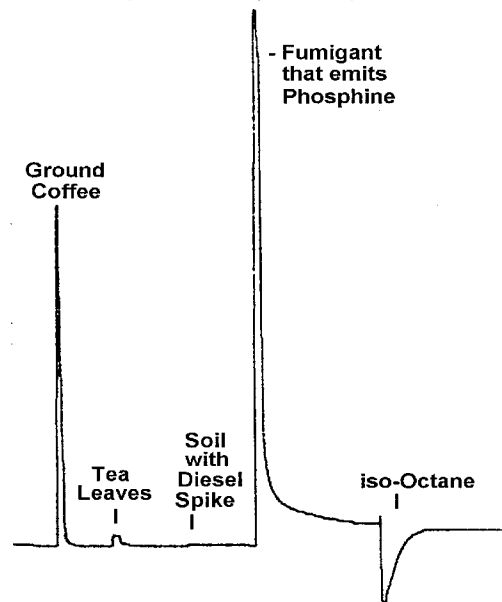


Figure 7

Figure 6. Schematic of NPD Transducer for Air samples.

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TID-1 Transducer for Samples in an Air stream.

TID-1 detection is the easiest to implement because it requires only Air as the operating gas. Figure 8 shows a schematic of the TID-1 Transducer used in this work. The transducer body incorporated a 1/8 inch Swagelok fitting for the Air inlet and a 1/4 inch Swagelok exit port. A 1/8 inch O.D. x 1/16 inch I.D. x 2 inch long tube was attached to the inlet to allow probing of vapors in small spaces. For the data of this report, a pump pulled an Air flow of 600 mL/min through the transducer.

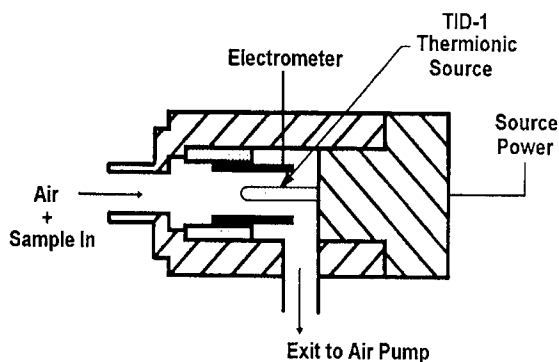
Figure 9 compares TID-1 signals for several different types of Halogenated samples. For these data as well as for previous FID and NPD data, the transducer inlet was exposed to the sample for a short period of time and the sample was then moved away. This procedure produced peak like signals such as the Iodine or Tribromo/Trichloro-phenol signals. However, exposure to vapors of Dichloromethane produced a longer term "tailing" of the signal peak indicating that this chlorinated sample had temporarily changed the charge emission characteristics of the TID-1 surface, and that effect lingered on for some time after the sample was removed. This is consistent with previous observations of TID-1 detection in GC applications. For sensing Volatile Chlorinated compounds, a TID-3 ion source which operates at a higher surface temperature provides much less tailing (i.e., faster response time).

We have known for some time that Iodine vapors produce an especially large TID-1 signal. From a consideration of the vapor pressure of Iodine at room temperature, we estimate that the TID-1 response in Figure 9 corresponded to an I₂ concentration in the range of 10 - 100 ppm in the incoming Air. Sniffing vapors emanating from a few crystals of Iodine in a vial has proven to be a simple way of determining the magnitude of ion source heating current to use in order to achieve a large TID-1 response.

The large TID-1 signal obtained from the Bromo/Chloro-phenol sample illustrated the good selectivity and sensitivity obtained for these compounds despite the fact that their concentrations in the sampled vapors were much less than the concentration of the more volatile Methanol.

Figures 10 and 11 compare TID-1 responses for vapors emanating from some food products. It was known from previous GC work that TID-1 ionization produced a good response to Vanillin, and that is consistent with the Coffee Bean data in Figure 10. Similarly, it was known previously that the compound Allyl Isothiocyanate (i.e., principle component of volatile oil of mustard) produced a large TID-1 response, and that is consistent with the comparison of Mustard samples in Figure 11.

TID-1 TRANSDUCER FOR SAMPLES IN AIR



TID-1 Transducer

Air = 600 mL/min

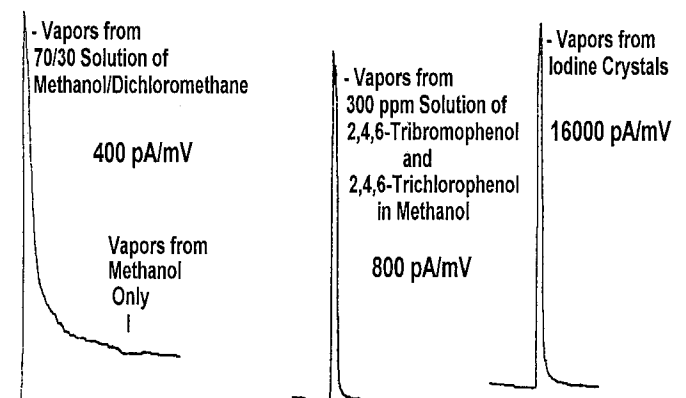


Figure 9.

Figure 8. Schematic of TID-1 Transducer for samples in an Air stream.

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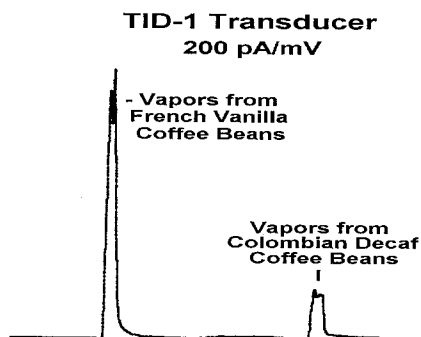


Figure 10.

Figure 12 shows a comparison of TID-1 responses to various fragrance essences obtained from Parfumerie Fragonard in France. This demonstrates TID-1 selectivity for detecting certain types of fragrances versus others. Unlike the FID or NPD, a TID-1 transducer is not destructive to the sample compound, so distinctive aromas can be sensed at the exit of the transducer. Also, it is possible to series couple a TID-1 transducer with an NPD or FID transducer to simultaneously generate 2 different detection signals from the same sample.

Summary. The FID, NPD, and TID-1 transducers described in this section were all self-heated and thermally insulated by fiberglass sleeving wrapped in a mummy-like fashion around the transducer body. Air flow through the transducers was provided by attaching a sampling pump to the exit port of the transducer. The magnitude of the Air flow rate through

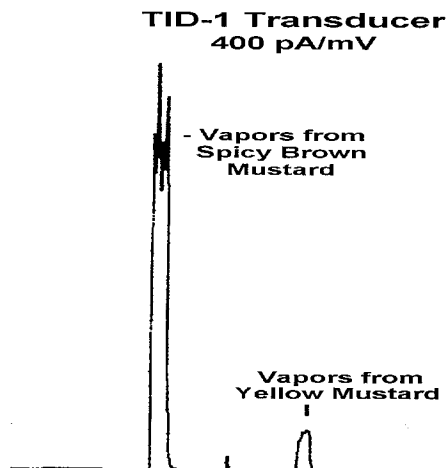


Figure 11.

the transducer was determined by the capacity of the sampling pump as well as by the upstream flow restrictions such as the size of the inlet tubing or packing of glass wool in the inlet. For the FID transducer, a 5 L/min pump (i.e., Laboport model from KNF) was used, while a 1.5 L/min pump (Schwarzer Prazision) was used with the NPD and TID-1 transducers. To eliminate Water condensation in the pump tubing, the FID transducer had a 1/4 inch Swagelok Tee attached to the transducer exit with a 1/4 x 1/8 inch tube port reducer attached at the tee location. This configuration provided an Air flow of 0.50 L/min through the transducer and 4.5 L/min purge at the transducer exit. The FID, NPD, or TID-1 transducer inlets were either tube or Swage type fittings that could be coupled to standard type gas connections if required.

Power and signal measuring electronics for the transducers was provided by a stand-alone DET Current Supply and a Keithley 6485 Picoammeter. The Current Supply provided both heating currents and polarization voltages for the FID Ignitor/Polarizer Probe, and the TID-1 and TID-2 Thermionic Ion Sources. FID and TID-1 detection used a polarization of - 45 V, while the NPD used a polarization of - 5 V.

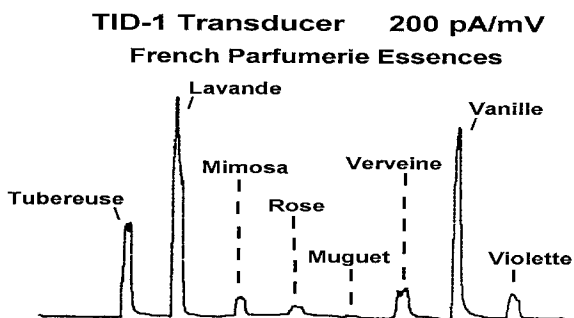
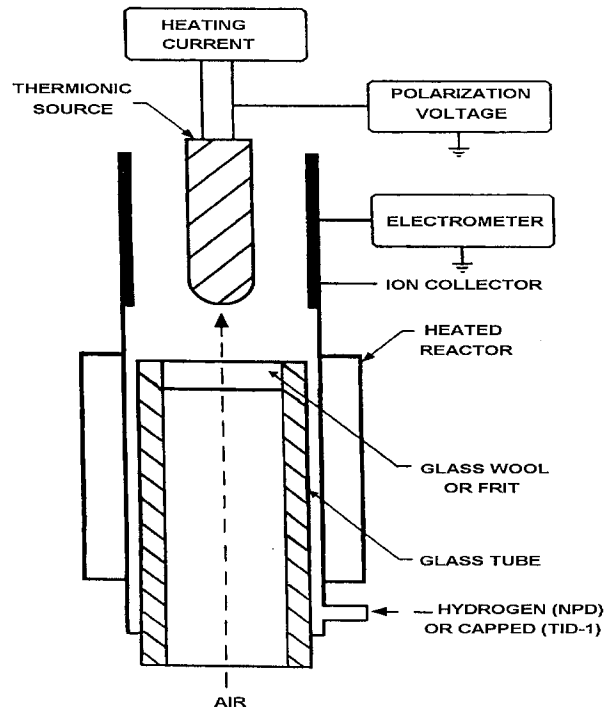


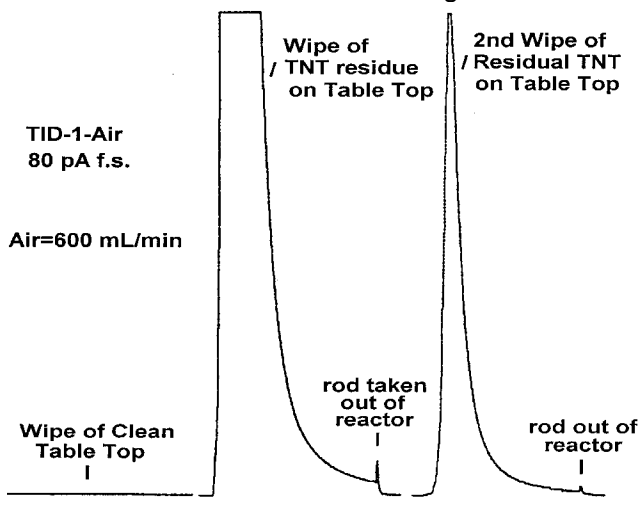
Figure 12.

Reactor Thermionic Ionization Analysis (RTIA) selective TID/NPD transducer screening of vapors evolved from THERMAL DESORPTION/OXIDATION of NONVOLATILE liquid residues or solid sample constituents

Background: Thermal desorption continues to be an increasingly popular means of preparing real world samples for subsequent analysis by gas chromatography. Among the many different types of GC detectors, TID and NPD detectors have the distinctive characteristic that they can provide chemical species selectivity using Air as the primary detector gas. Consequently, these detectors are well suited to non-GC chemical screening applications where the gas environment is simply ambient Air drawn through a TID/NPD transducer by a sampling pump. In an **RTIA** configuration, such a transducer is preceded by a heated reactor chamber into which are inserted solid samples packed into a glass tube or liquid sample residues on a ceramic rod. The TID or NPD transducer provides selective responses to vapors evolved from thermal desorption and/or oxidation of the samples. At low reactor temperatures, thermal desorption usually accounts for most detected signals, while at high temperatures oxidative sample decomposition products often provide large signals. Good examples of the oxidative detection processes are large TID-1 signals from oxidation of sugars and proteins. For TID-1 and TID-3 thermionic detection, only Air is required as the operating gas, while for NPD a small flow of Hydrogen has to also be introduced into the incoming Air.



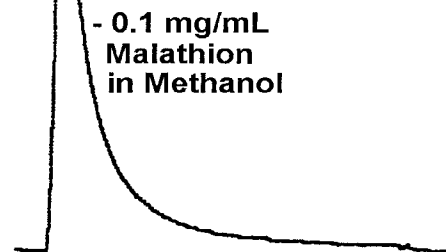
i-Propanol Wipe Transfer of Surface Residue to 1/16 in. Ceramic Rod - Rod inserted into 140 deg C Reactor



Malathion Residue on Quartz Rod

NPD, 160 pA f.s., Hydr.=4.3, Air=200

rod dipped into sample -
 Methanol evaporated -
 rod residue inserted into
 140deg C reactor



dried TNT residue on a table top wiped with an isopropanol pad and transferred to ceramic rod.

small quartz rod dipped into a 0.1mg/mL solution of Malathion in Methanol.

DET

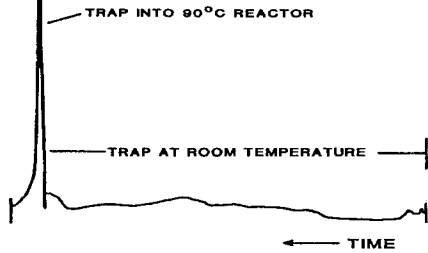
innovations in chemical detection

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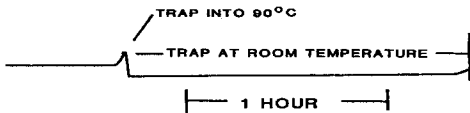
Examples: RTIA/TID-1 Analyses

TRAP-DESORB-DETECT

AIR SAMPLING THROUGH CARBOTRAP
AMBIENT WITH TRACE TRICHLOROETHANE

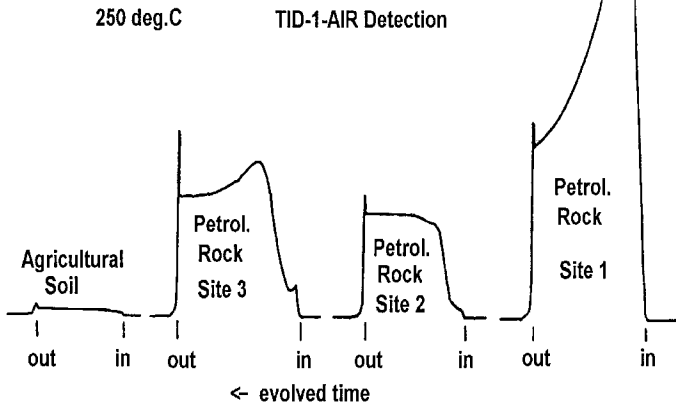


AMBIENT WITHOUT TRICHLOROETHANE



RTIA/TID-1. 1 L/min ambient Air - 1 hour through unheated trap, then trap into 90°C reactor.

Petroleum Source Rock from 3 Different Geological Sites
4mm dia. x 13mm Column of Sample into Hot RTIA Reactor

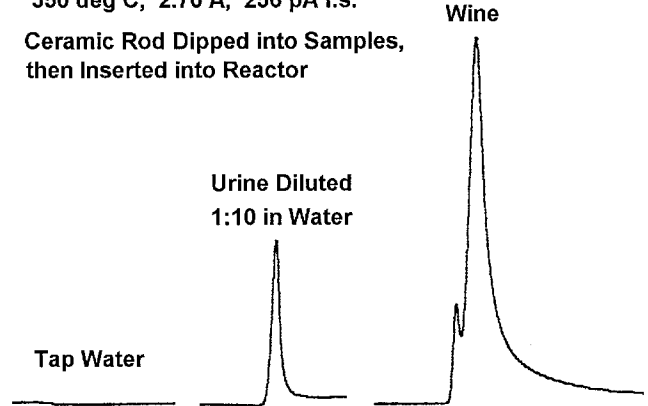


crushed rock/soil loosely packed into 1/2 inch long column inside 6mm OD x 4mm ID glass tube - tube inserted and sealed into reactor so incoming Air flowed through tube ID and the samples - sample tubes inserted/removed as indicated - peak in signals followed by decay indicates constituent being volatilized and removed - steady signal plateau indicates constituent vapor pressure, but no volatilization/removal.

DESORB/OXIDIZE & DETECT

350 deg C, 2.70 A, 256 pA f.s.

Ceramic Rod Dipped into Samples,
then Inserted into Reactor

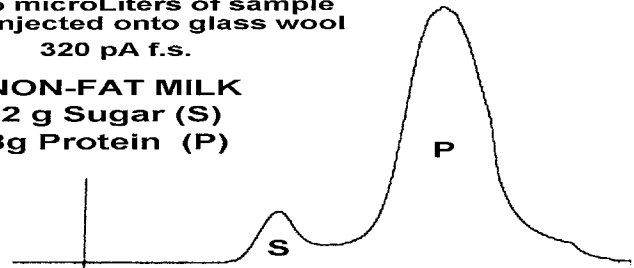


water based liquid samples - ceramic sample rod moist with both water and residual organic material when inserted into the reactor - signals mostly due to oxidation products of the organics.

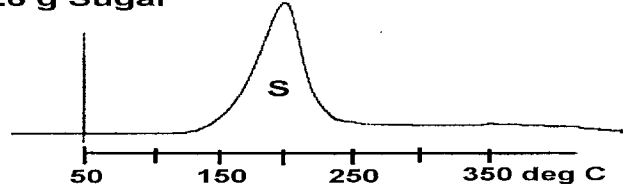
RAMPED TEMPERATURE OXIDIZE & DETECT

5 microLiters of sample
injected onto glass wool
320 pA f.s.

NON-FAT MILK
12 g Sugar (S)
8g Protein (P)

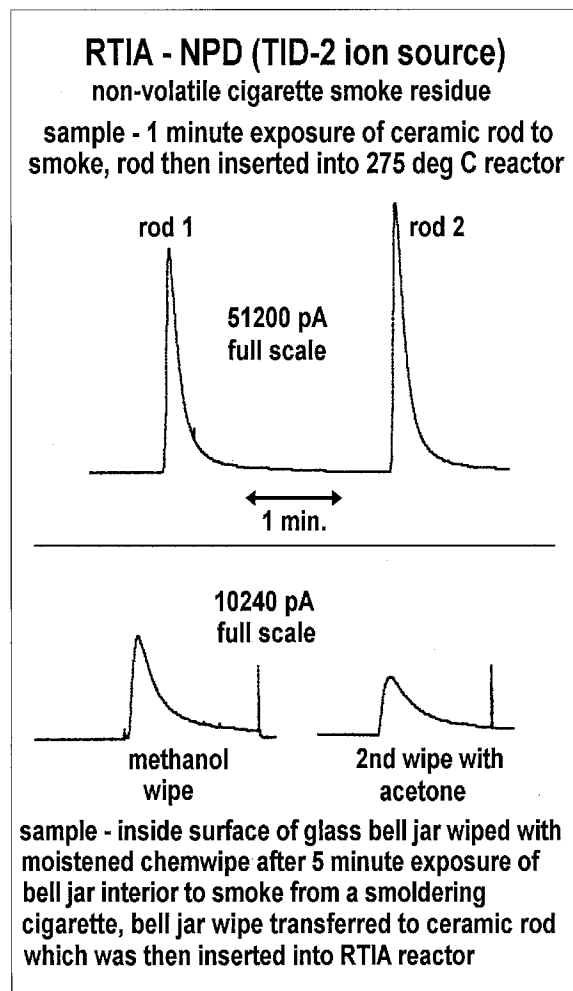
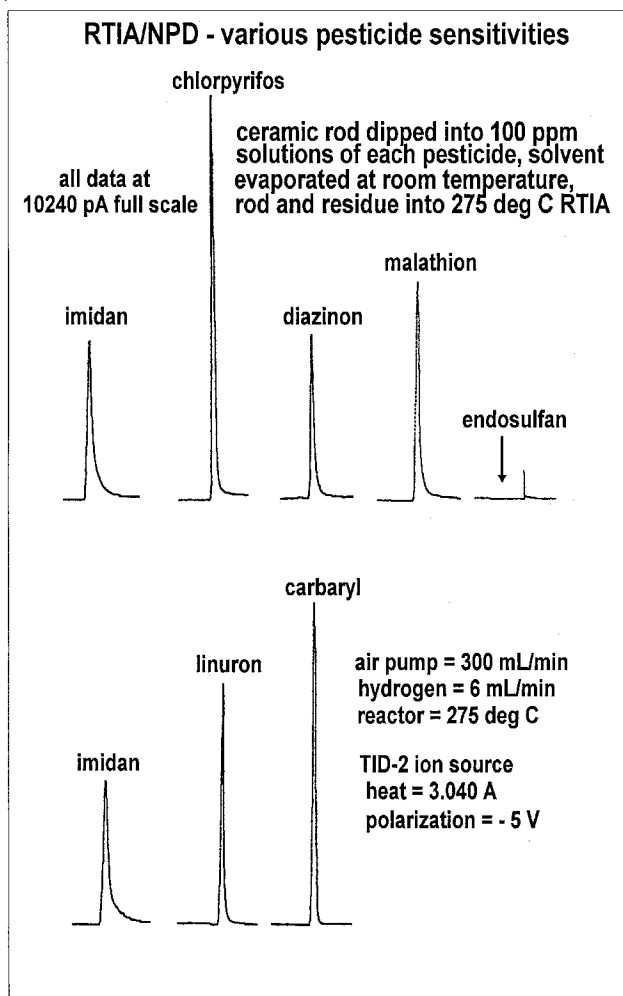


FLAVORED JUICE DRINK
26 g Sugar



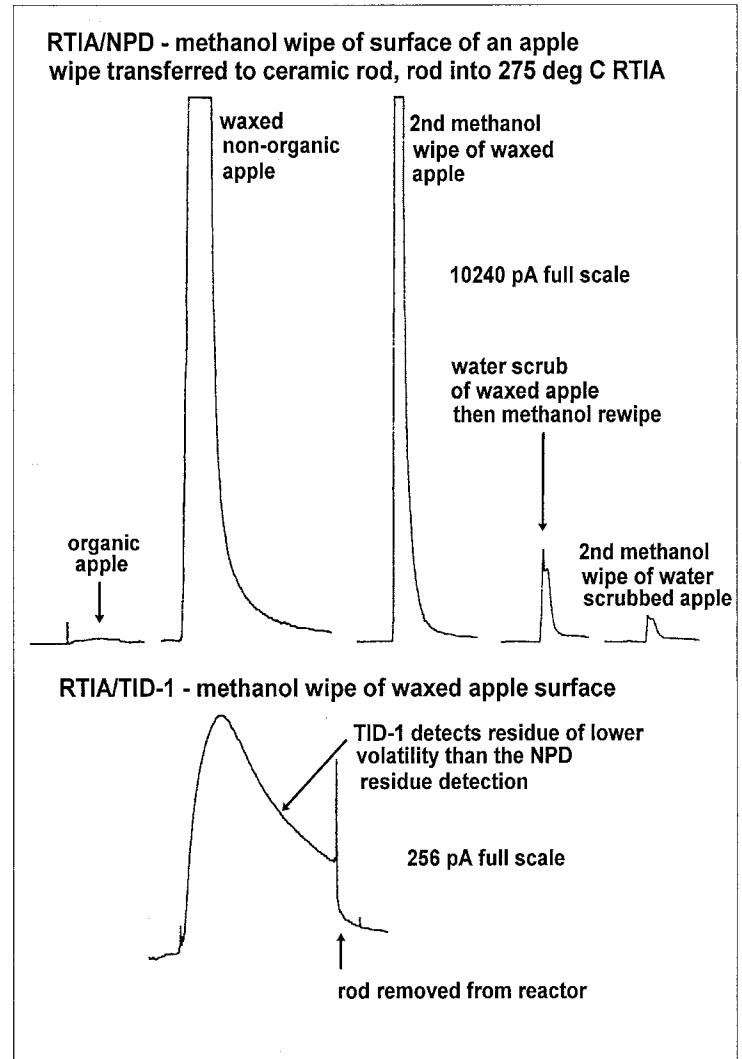
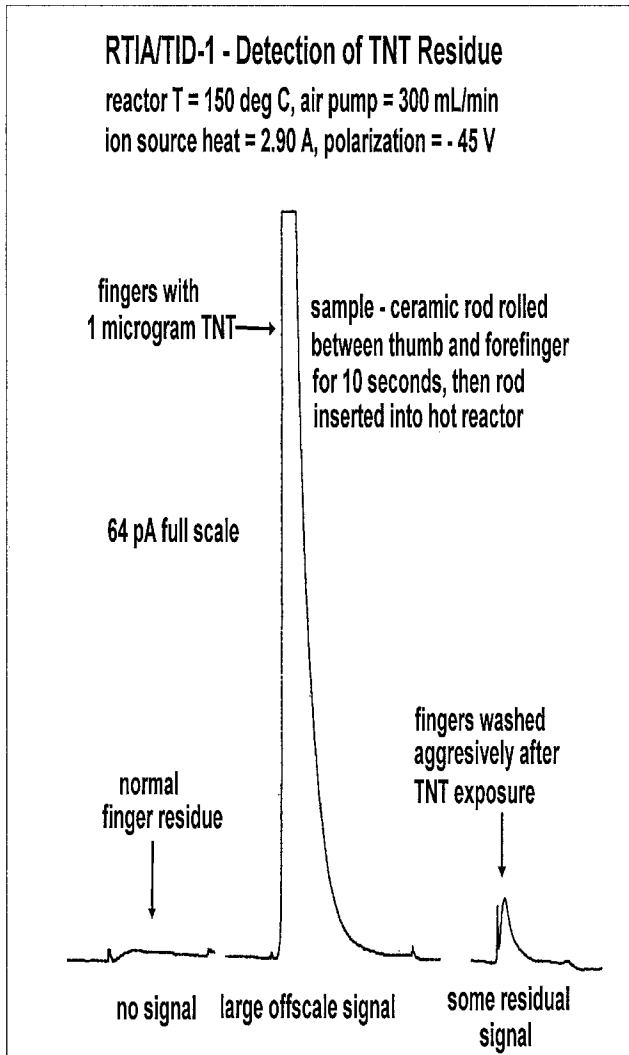
variation of the RTIA technique - TID-1 detector mounted onto a GC - 2 inch long 6mm OD x 4mm ID open end glass tube packed with glass wool located inside GC oven and connected to detector inlet - sample pump at detector exit pulled Air through the system - liquid samples injected onto glass wool - column oven temperature programmed to provide profile of oxidation product signals vs. temperature.

Different Methods of Acquiring Non-Volatile Sample Residues on a Ceramic Rod for Subsequent Selective Screening by the RTIA Method



Tip of 1/16 inch diameter ceramic rod dipped into solution of non-volatile pesticides in a volatile solvent like Methanol. Solvent evaporated, then rod inserted into RTIA reactor for detection of selected vapors with a Nitrogen-Phosphorus Detector. RTIA/NPD detected N and P pesticide residues, but not Endosulfan which contained lots of Cl and no N or P. Samples could be liquid extracts of farm soil, plantings, or crops. Other type TID transducers can provide similar selective detection of Halogenate and Triazine pesticide residues.

“Third Hand Smoke”. RTIA/NPD detection of non-volatile residues deposited on surfaces exposed to Second Hand Smoke. Top data illustrate signals from smoke residues deposited directly onto tips of 2 ceramic rods. Bottom data illustrate Methanol and Acetone wipes of a surface exposed to Second Hand Smoke, with the wipes subsequently transferred to the tip of a ceramic rod. Data demonstrate simple means of monitoring ambient environments for exposure to potentially toxic non-volatile chemicals in those environments.



RTIA/TID-1 detection of a TNT trace residue on a finger. Sampling procedure consisted of rolling the tip of a ceramic rod between the thumb and forefinger, and then inserting the rod into the RTIA reactor equipped with a TID-1 detector which has exceptional sensitivity for Nitro explosives like TNT. Data demonstrate that some detectable residue is still left even after vigorous washing of hands.

RTIA/NPD & RTIA/TID-1 detection of an Apple's Wax coating. Sharp rise and fall of NPD signal corresponded to a more volatile component than the slower oxidizing components detected by TID-1. The TID-1 signal was likely due to CH_2 groups in the wax compounds. The NPD signal is likely associated with a preservative like Diphenylamine which is widely used to prevent apple discoloration, and these data illustrate how difficult it is to remove this preservative by simple washing. This Wipe/Transfer RTIA technique is a simple means of screening food products.

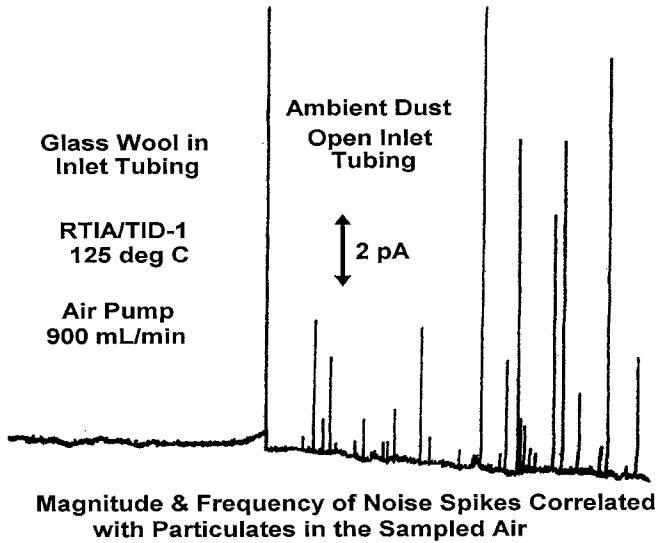
DET

innovations in chemical detection

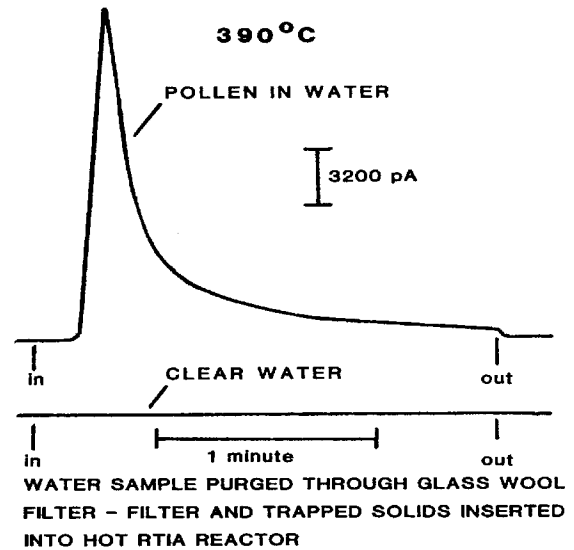
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MISCELLANEOUS RTIA/TID-1 DATA EXAMPLES

PARTICULATE DETECTION



TRAP-DESORB/OXIDIZE-DETECT

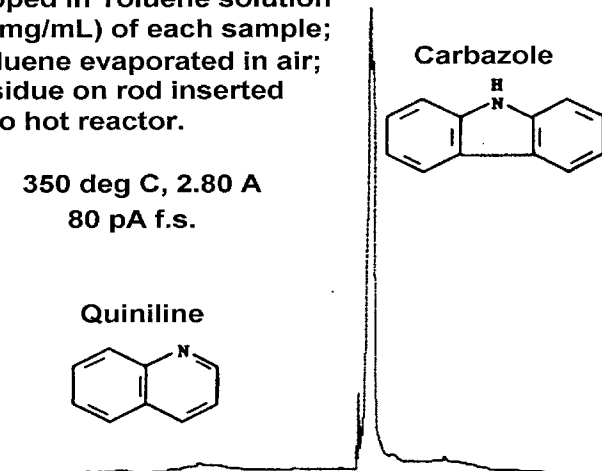
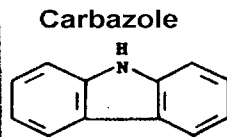
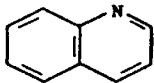


DESORB & DETECT

1/16 in. dia. ceramic rod dipped in Toluene solution (5 mg/mL) of each sample; Toluene evaporated in air; residue on rod inserted into hot reactor.

350 deg C, 2.80 A
80 pA f.s.

Quiniline

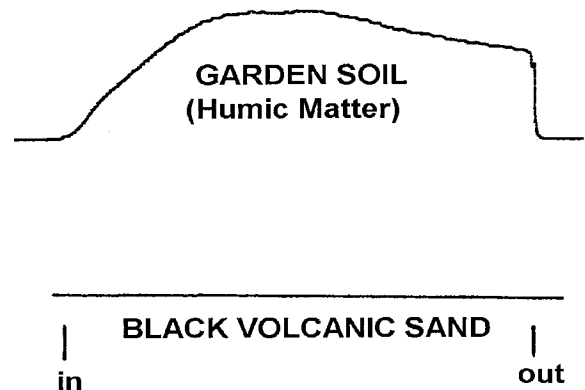


good illustration of TID-1 selectivity for compounds with the Pyrrole group but not the Pyridine group

DESORB/OXIDIZE-DETECT

350 deg C, 2.70 A, 25600 pA f.s.

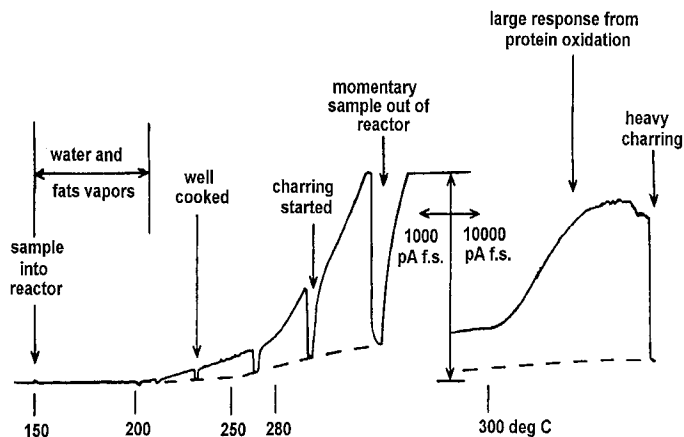
50 mg SOIL SAMPLES into REACTOR



comparison of soil samples with and without fertile nutrients

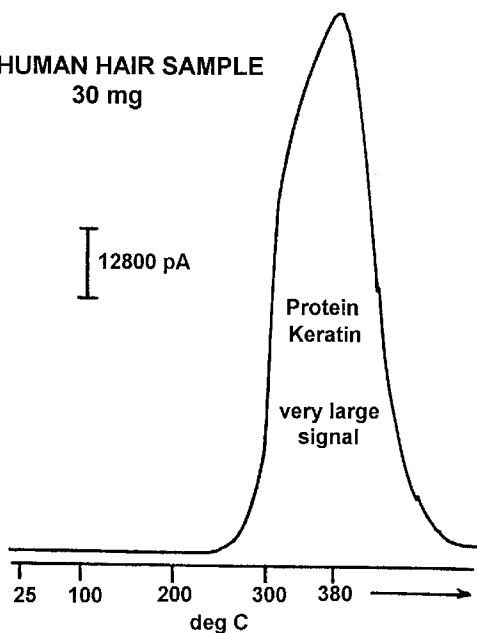
MISCELLANEOUS RTIA/TID-1 DATA EXAMPLES

COOKING OF RAW HAMBURGER

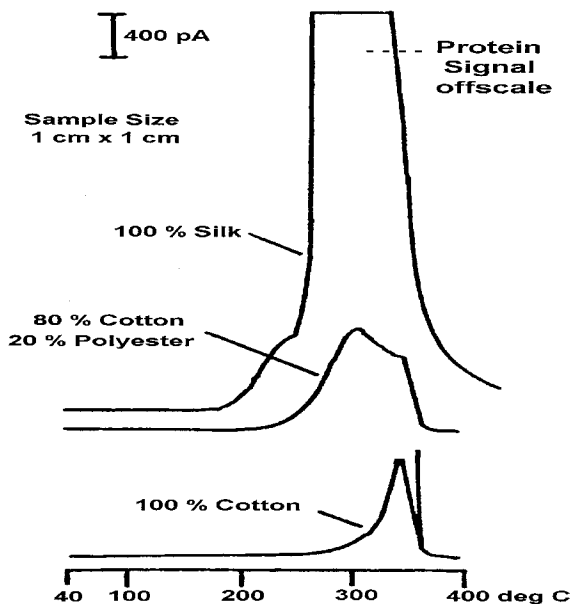


RTIA monitoring of Hamburger cooking - (good demonstration of ability to tolerate complex sample matrices) - raw Hamburger packed into glass sample tube - tube inserted into RTIA reactor initially set at 150 deg C (302 deg F) - water & fats vapors allowed to evolve - then reactor temperature increased in increments as shown - glass sample tube removed periodically to allow visual inspection of cooked state of the hamburger - very large TID-1 signal to vapors produced by oxidation of Hamburger Protein.

HUMAN HAIR SAMPLE 30 mg



FABRICS



Examples of very large TID-1 responses to Thermal Oxidation Products of Proteins like Human Hair and Silk fabric - GC oven converted to RTIA type analysis with temperature program for sample packed in glass tube, TID-1 detector, and exit pump pulling ambient Air through sample and detector.

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DET TID/NPD TRANSDUCER CONNECTED TO A HEATED INLET REACTOR CHAMBER.

This assembly is used in an RTIA module (Reactor Thermionic Ionization Analyzer) with a sample pump at the transducer exit pulling ambient Air through the Reactor/Transducer. The Transducer provides selective screening of vapors produced from Thermal Desorption and/or Oxidation of Non-Volatile constituents in Liquid or Solid samples. Residues deposited onto a 1/16 inch diameter ceramic rod are one method of introducing samples into the hot Reactor/Transducer.



DET CURRENT SUPPLY, KEITHLEY ELECTROMETER,
AND RTIA MODULE WITH AIR SAMPLE PUMP

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STAND-ALONE TRANSDUCER EXAMPLE



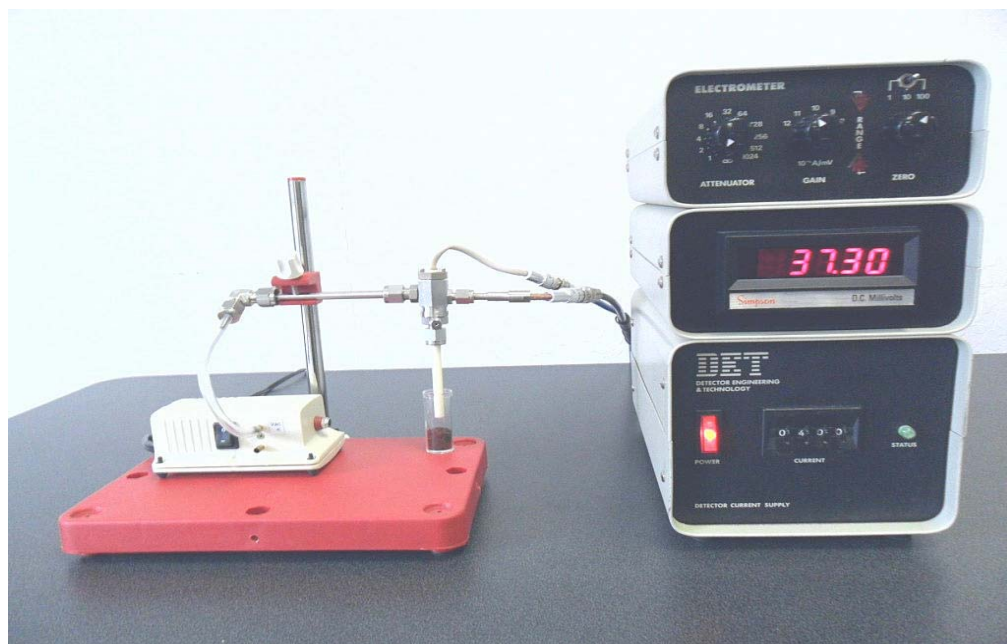
1.) Air Sample Pump Attached to Exit Port of TID/NPD Transducer:

Real Time TID Screening and Monitoring of Selected Vapors Evolving in Air Head Space from Solid or Liquid Samples (e.g., Acetic and other Volatile Acids from Food Products; TNT, 2,4-DiNitrotoluene Explosives or Nitro Pesticide Residues in Soil; Halogenated Vapors like Cl_2 or I_2 ; Flavor and Aroma Compounds like Allyl Isothiocyanate and Vanillin; Alcohols produced in Fermentation Processes; Sugar and Protein Decomposition Products produced in Food Cooking Processes; etc.)

TID/NPD Selectivity of Detection Determined by Choice of the Type of Ceramic Ion Source.

NPD Selectivity Achieved by also Adding Small Flow of Hydrogen Through 1/16 inch Swage Tee at Inlet.

(Transducer hardware is available with different choices of Swage or Tube Inlet and Outlet fittings for ease of attachment to various types of flowing gas streams.)



Transducer Connected to Air Sample Pump and Tethered to an Electronic Control Module Containing a Current Supply for Heating and Polarizing Ion Sources, and Electrometer for Measuring Signals.

(Note: A Keithley 6485 Picoammeter is currently used as the Electrometer in place of the top 2 sections of the electronics shown.)

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EXAMPLES OF DET HARDWARE STRUCTURES



DET NPD hardware for custom fit onto a Bendix Process GC FID base. DET structure is made from 0.75 inch hexagonal stainless steel stock, approximately 1.50 inches tall. DET structure fits inside Bendix detector housing with ion source power wiring (2 wires) and electrometer signal wiring (1 wire) connecting to terminals in base of Bendix detector housing. Stand-alone DET Current Supply used to power ion sources, and Bendix negative ion electrometer used for signal measurement.



DET Transducer tower structure with a 3/4 inch Swagelok inlet fitting. Gas outlet is a 1/4 inch Swagelok fitting. Various size inlet/outlet fittings are available in either a Swage or tube type configuration. Minimum size inlet/outlet is a 1/16 inch Swage fitting or a 1/8 inch diameter tube.



DET Transducer tower with a 1/8 inch Swage inlet and a 1/16 inch Swage inlet tee port, and a 1/4 inch Swage outlet.

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EXAMPLES OF DET TRANSDUCER TOWER CONFIGURATIONS



1/8 inch Swage in, 1/8 inch tube out, with tower mounting flange



1/8 inch Swage in, 1/8 inch tube out, with tower and mounting flange attached to a small Aluminum heater block.



1/4 inch Tube in, 1/4 inch Swage out



3/4 inch Swage in, 1/4 inch Swage out