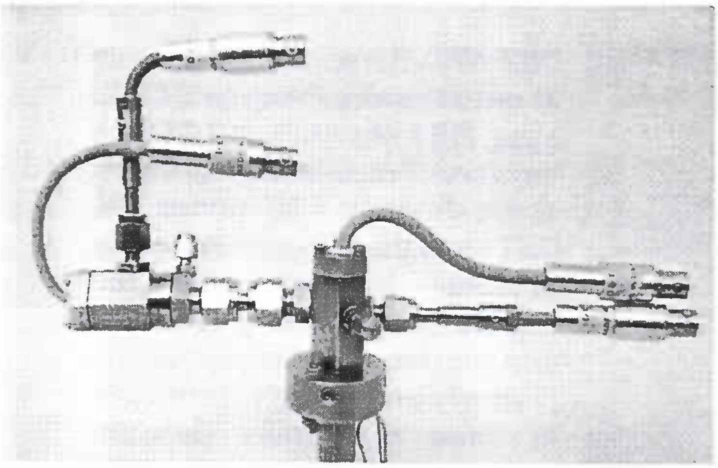
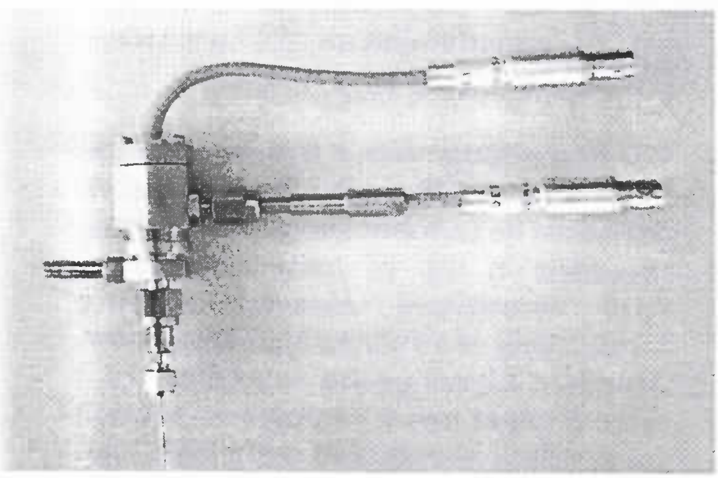
DET
innovations in chemical detection

CHROMalytic TECHnology Pty Ltd +61(0)3 9762 2034
Australian Distributors; Importers & Manufacturers

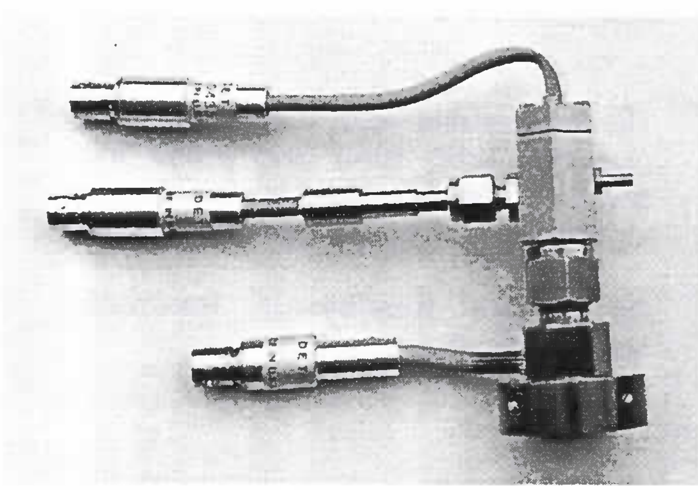
EXAMPLES OF DET HARDWARE STRUCTURES



Tandem TID/FID hardware for custom fit onto Varian GCs. Two gas lines in Varian detector base supply gas environment for TID stage of detection. Third gas inlet after TID detection supplies Hydrogen flow through ceramic jet for second stage FID detection. Fourth gas connection at FID inlet supplies Air flow about outer periphery of FID jet.

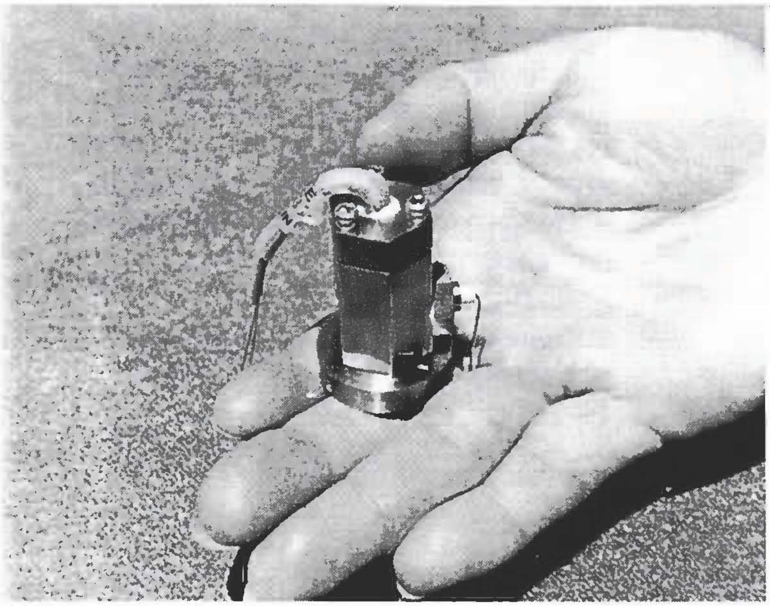


FID transducer for samples in an incoming Air stream. 1/16 inch Swage inlet provides Hydrogen flow through a ceramic lined jet. 1/4 inch tube inlet provides sample and Air flow about outer periphery of FID flame jet.

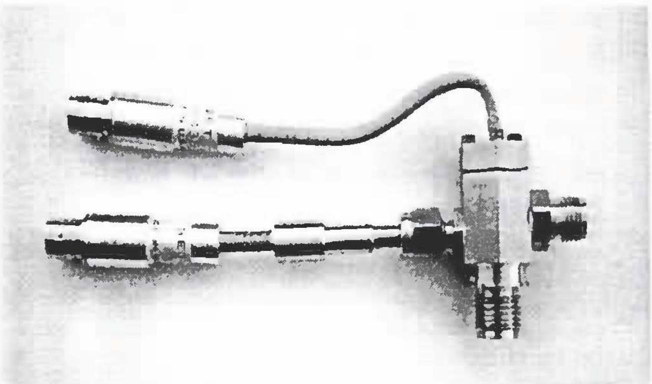


Flame Thermionic Ionization Detector (FTID) structure that fits onto Varian GC detector base. Samples decomposed in flame at short ceramic jet. Polarization voltage supplied at bottom of detector drives flame ions to the surrounding detector wall. Thermionic transducer attached downstream re-ionizes neutral Halogen and/or Nitrogen combustion products from the flame.

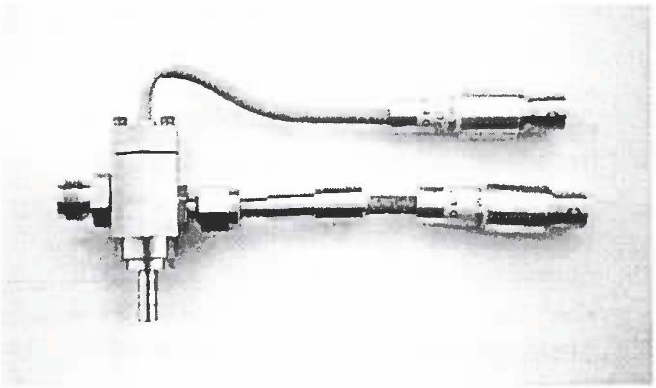
EXAMPLES OF DET HARDWARE STRUCTURES



DET NPD hardware for custom fit onto Bendix Process GC FID base. DET structure is 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.

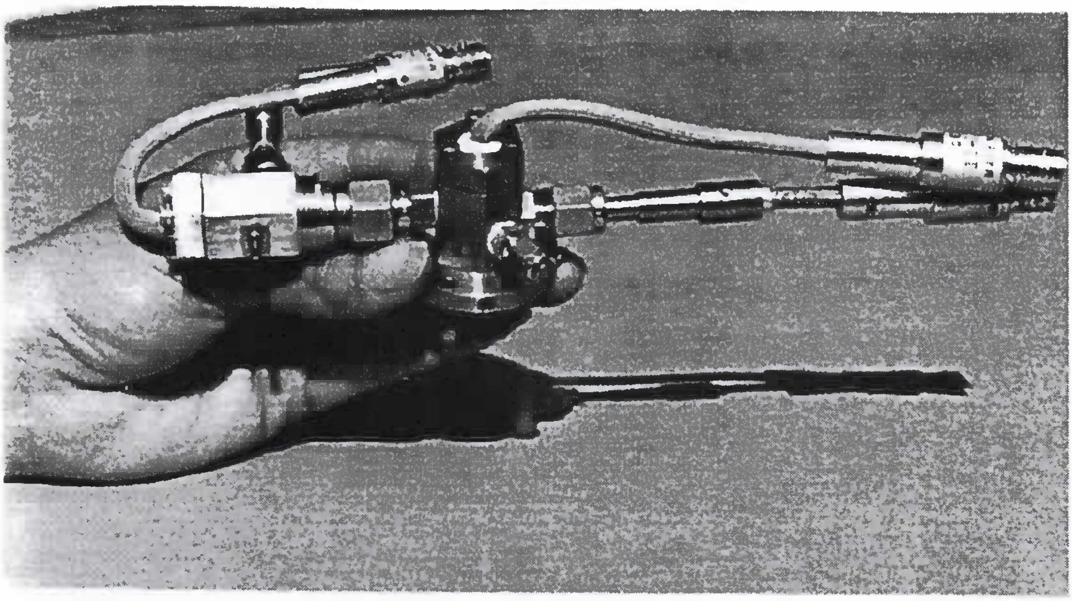


TID Transducer with 0.375 inch Swage inlet and 0.250 inch Swage outlet. Standard hexagonal flanged ion source mounted in top of transducer tower with fiberglass sleeved cabling terminating in a Twinex type connector. Standard signal probe extending from side of tower has a flexible mid section for bending as required and a BNC type connector for cabling to a negative ion electrometer.

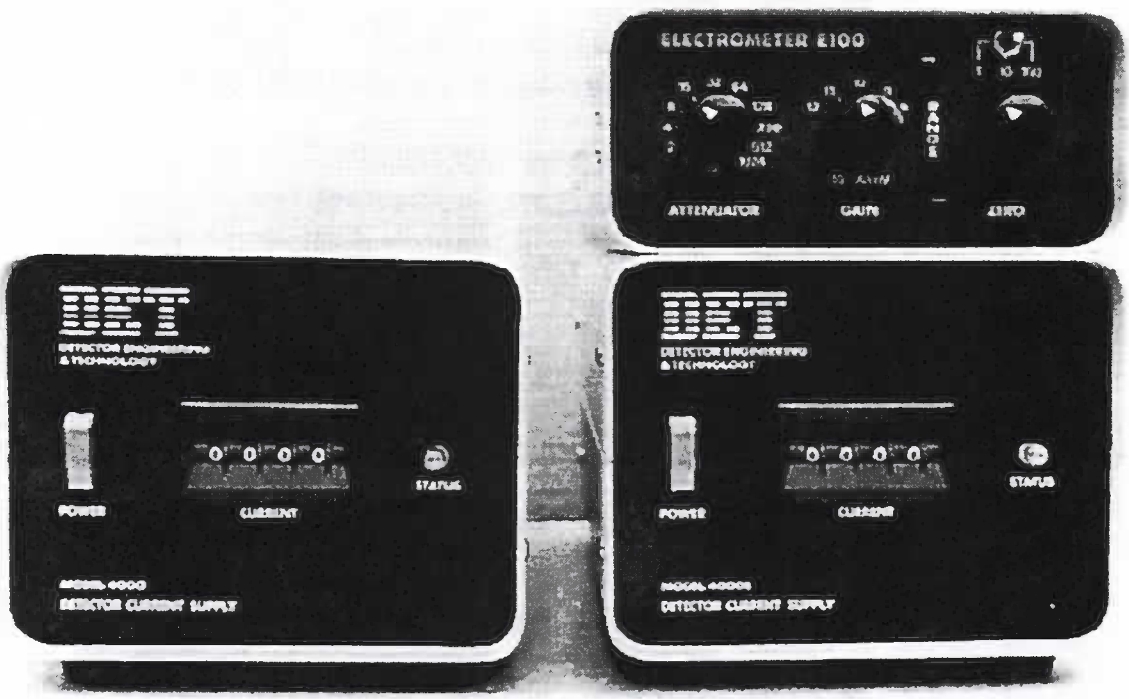


TID Transducer with 0.250 inch outer diameter inlet tube and 0.250 inch Swage outlet. Standard ion source and signal connections as described above.

Other size tube/Swage inlet/outlet fittings are also possible.



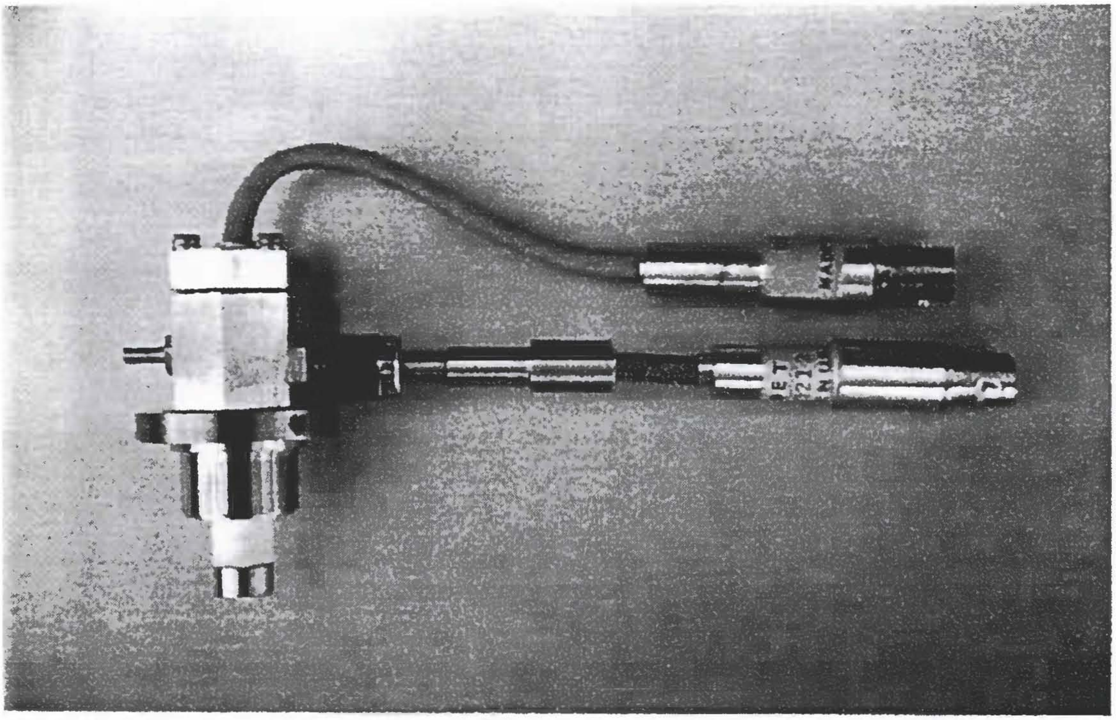
TANDEM TID - 2 simultaneous signals



DETECTOR CURRENT SUPPLY

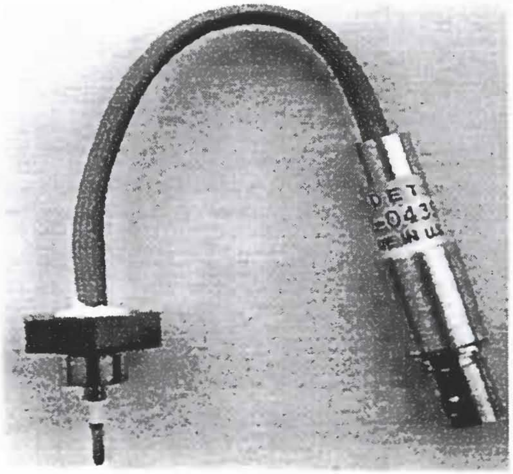
COMBINED CURRENT SUPPLY AND ELECTROMETER

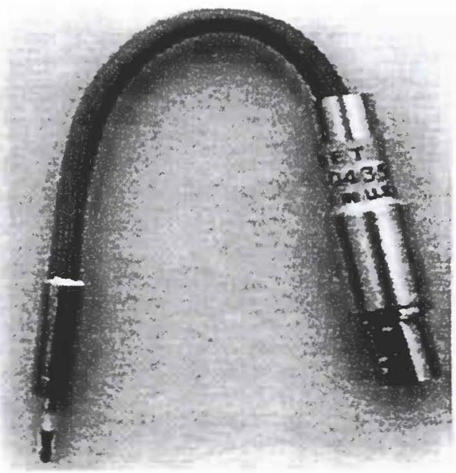
DET innovations in chemical detection



DET NPD/TID/REMOTE FID HARDWARE
MOUNTS ONTO AGILENT 6890 FID BASE OR HP 5890 FID/NPD BASE

THERMIONIC IONIZATION SOURCES
(AVAILABLE WITH OR WITHOUT ELECTRICAL CONNECTOR)



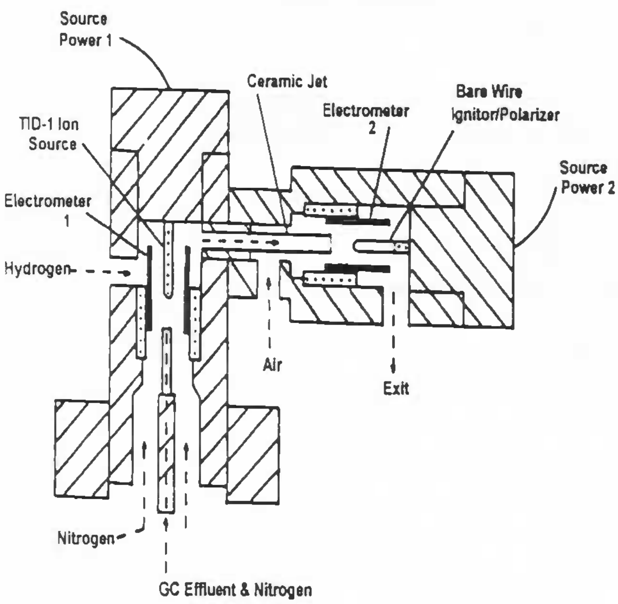


STANDARD HEXAGONAL FLANGE MOUNTING FITS ALL DET HARDWARE AND AGILENT 6890 NPD HARDWARE

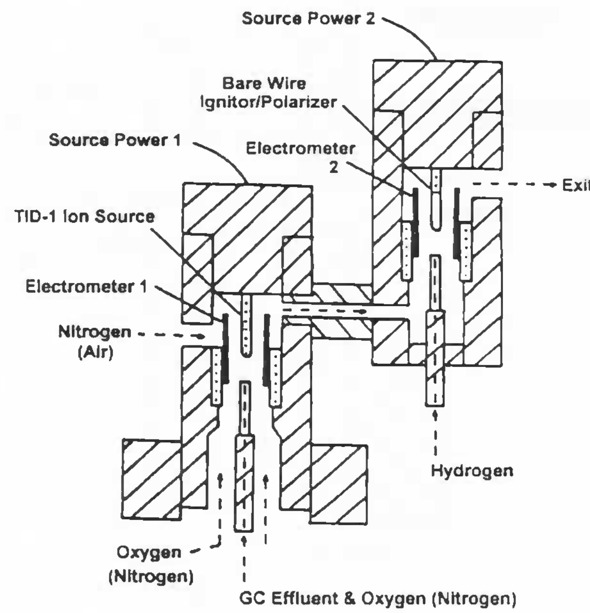
1/4 INCH TUBE MOUNTING FOR CUSTOM APPLICATIONS. USED IN THERMO-FINNIGAN AND SRI GCs.

TANDEM TID/FID: SIMULTANEOUS TID and FID SIGNALS

Tandem TID-1-Nitrogen/FID



TANDEM TID-1-Oxygen (Nitrogen)/FID

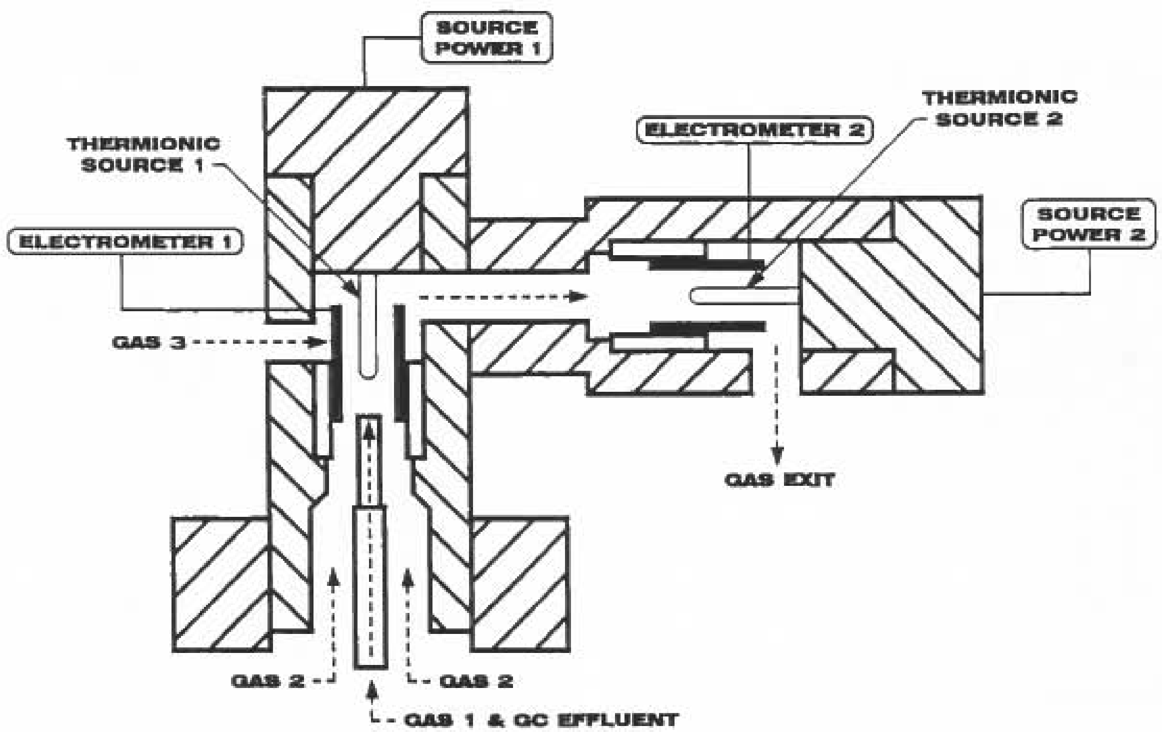


Equipment:

In a **TANDEM TID/FID**, two independently controlled thermionic and flame ionization detection stages are combined in a series combination. The first stage is a **MODIFIED TID/FID** tower (Varian GC models) or a **MODIFIED REMOTE FID** tower (Agilent 6890 and HP 5890 GC models) that mounts onto the existing FID base on the GC. (The mounting configuration illustrated above is that which fits Varian instruments.) The second stage is either an **FID TRANSDUCER/JET ASSEMBLY** for the case where the first stage effluent is carried into the FID in an inert gas stream, or an **FID/AIR SAMPLE TRANSDUCER** for the case where the first stage effluent is carried into the FID in an oxidizing gas stream. In either case, two different detector gases can be supplied through the lines in the detector base that normally supply "H_2" and "Air" to an FID. A third gas inlet in the modified first stage tower provides an additional gas flow between the two stages. The second stage **FID TRANSDUCER/JET ASSEMBLY** includes a fourth gas inlet for Air to sweep the outer periphery of the FID flame jet. The second stage **FID/AIR SAMPLE TRANSDUCER** includes a fourth gas inlet for H_2 to the flame jet. A ceramic coated thermionic ion source such as the **TID-1** or **TID-3** is normally used in the first stage, while a bare wire **FID Probe** is used as the flame ignitor/polarizer in the second stage. Each stage of detection requires a separate heating current and polarization electronic module for powering the ion source/FID probe. The simultaneous TID and FID signals from the two stages require two negative ion electrometers for measurement.

DET innovations in chemical detection

TANDEM TID: 2 SIMULTANEOUS SIGNALS, many signal combinations are possible



Equipment:

In a **TANDEM TID**, two independently controlled thermionic ionization detection stages are combined in a series configuration. The first stage is a **MODIFIED TID/FID** tower (Varian GC models) or a **MODIFIED REMOTE FID** tower (Agilent 6890 and HP 5890 GC models) that mounts onto the existing FID base on the GC. The second stage is a **TID TRANSDUCER** that attaches to the exit port of the first stage. Two different detector gases can be supplied through the lines in the detector base which normally supply "H_2" and "air" to an FID. A third gas inlet in the modified first stage tower provides an additional detector gas flow between the two stages. The two detection stages can be easily decoupled to allow separate operation of each structure. The first detection stage can be purchased separately, and the second stage may be added later as needed.

Each stage requires a thermionic source or FID probe, and their separate heating current and polarization electronics. The simultaneous signals from the two stages require two negative ion electrometers for measurement.

Response:

Many different tandem signal combinations are possible, depending on the type of thermionic sources/FID probe used and the composition of detector gases supplied. Some possibilities are as follows:

TID-1-N_2/HWCID - simultaneous detection of oxygenates and hydrocarbons in gasolines;

TID-1-Air/NPD - simultaneous detection of organochlorine and nitrogen/phosphorus pesticides;

FID/FTID-2 - simultaneous detection of hydrocarbons and high concentration halogenates;

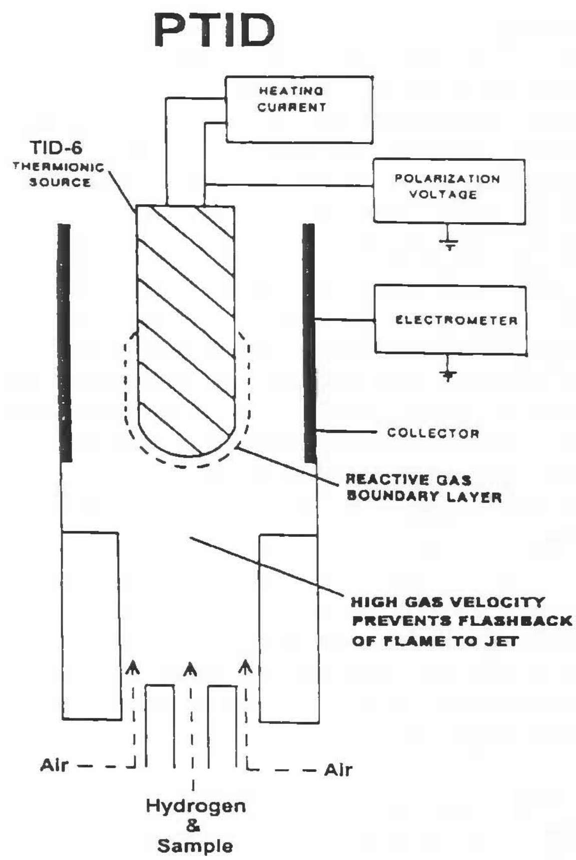
PTID: (Phosphorus Thermionic Ionization) selective detection and very large signals for P with suppressed N response.

Equipment:

This detection mode uses a **TID-6** type thermionic source mounted in a **PTID Tower**. The **PTID** tower structure is similar to a Remote FID Tower in positioning the thermionic source several centimeters downstream of the jet. However, the **PTID Tower** contains a reduced internal diameter for high gas velocity to prevent flame front flashback from the hot source to the jet. This allows higher Hydrogen and Air gas flows to be used than are possible with an NPD. In the PTID, typical gas flows are H_2 = 20 - 30 mL/min, and Air = 250 - 500 mL/min. The thermionic source is heated by a constant current supply and is polarized at - 15 V with respect to the collector. During operation, the surface temperature of the source is maintained in the range of 600 - 800°C which produces a visible orange glow.

Principle:

This mode uses a thermionic source of high work function operated in an FID-like H_2/air environment. However, unlike an FID, an internal flow restrictor prevents flame front flashback to form a self sustaining flame at the jet orifice where H_2 and Air are initially mixed together. Instead, an ignited, chemically active gas boundary layer is maintained about the hot source surface similar to an NPD. Because of the higher H_2 and Air, this PTID boundary layer has a much higher concentration of chemically active radical species. NPD thermionic sources do not hold up well in this harsher environment, so this mode of detection requires a more durable ceramic source surface. Like an NPD, sample compounds are decomposed in the gaseous boundary layer, and P compounds form decomposition products which are converted with high efficiency to gas phase negative ions by extracting electrons from the thermionic surface.



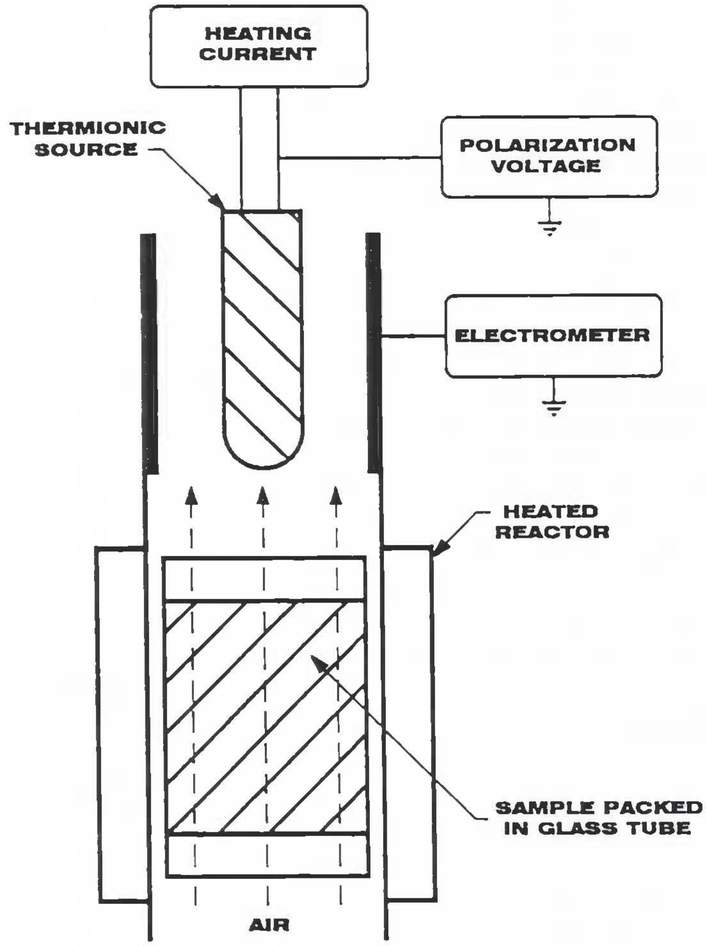
Response:

PTID response to P compounds is generally more than 10 times larger than the corresponding response of an NPD. However, the background and noise are also larger, so detectivity is comparable to an NPD (0.07 pg P/sec). The dynamic range of response of a PTID exceeds 5 orders of magnitude, and it has excellent selectivity vs. hydrocarbons, as well as vs. N, O, Cl, Br, S, and Si compounds.

RTIA: REACTOR THERMIONIC IONIZATION ANALYZER

Equipment:

RTIA modules are stand-alone units containing a thermionic ionization transducer preceded by a heated reactor chamber. Each module has a thermally insulated, temperature controlled (50-400°C) transducer and reactor, and supporting gas flow control elements or an air sample pump as needed. The transducer response is determined by the type of thermionic source used, and the type of gases supplied. Available modes include **TID-1-Air(N_2)**, **TID-3-Air(N_2)**, or **TID-2-H_2/Air** (i.e., NPD). TID-1-Air is an especially simple configuration in which the operating gas may be ambient air drawn in by a pump attached to the module exit port. Power and signal measurement for the transducer is provided by a stand-alone **CURRENT SUPPLY/ELECTROMETER** module.



Applications:

The RTIA selectively detects electronegative or NP vapors which thermally evolve from solid or liquid samples. Applications include a direct inject-vaporize procedure for liquid samples; a desorb-detect procedure for solid samples; and a trap-desorb-detect procedure for vapors in ambient air. The TID-1-Air mode detects nitrogen oxides and halogen/halogen oxides evolved in the thermal oxidation of food products, fabricating materials, oil bearing source rocks, and contaminated soil/water samples.

FID & HWCID & CFID: universal response to ALL ORGANICS

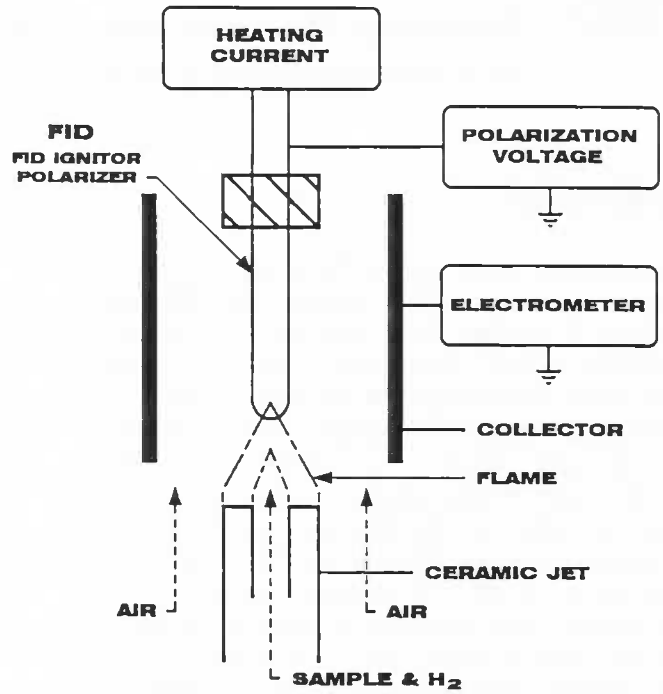
Equipment:

The FID mode uses an **FID Probe** consisting of a bare loop of wire. The Probe is mounted in a **TID/FID** tower which positions it in close proximity to an unpolarized flame jet. The flame jet in this detector is a high purity alumina ceramic (0.062 in. O.D. x 0.031 in. I.D.) which withstands long term operation without deteriorating oxidation, and which can operate at very high temperatures without excessive thermionic electron emission. The FID Probe functions as both flame ignitor and polarizer. H_2 and air are the detector gases, and a self-sustaining flame burns at the jet. The FID Probe is also used in an optional **FID Transducer/JET Assembly** for the second stage of a **Tandem TID** tower configuration. The FID Probe is also used in an exclusive **HWCID** (Hot Wire Combustion Ionization) detection mode to provide jet-less FID type responses using a **TID Transducer** second stage for the Tandem TID tower. In the HWCID, a flame-like environment is maintained by continuously supplying heating current to the probe wire. With standard DET electronics, the FID Probe is normally polarized at -45 Volts, and negative ions are collected. On some GCs, the FID Probe can also be connected to existing positive polarizing voltages in order to collect positive rather than negative ions.

For the **CFID** mode (Catalytic Flame Ionization), a CFID type thermionic source is used instead of the FID Probe. As in the FID, a self-sustaining flame burns at the jet. The CFID source is connected to polarizing and heating current electronics, so the source surface can be electrically heated as well as flame heated. The source in this mode serves the threefold purpose of flame ignitor, polarizer, and catalytic combustion modifier. The CFID source is typically polarized at -5 Volts, and negative ions are collected.

Principle:

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,



$$CH + O \Rightarrow CHO^+ + e^-,$$

where two neutral species combine to yield equal concentrations of positive and negative charged species in the gas phase. In addition to the gas phase ionization of an FID, the CFID provides a secondary thermionic ionization mechanism on the CFID source surface. The secondary mechanism selectively affects responses to compounds containing heteroatoms such as Cl and P. The relative magnitude of the secondary ionization is controlled by varying the heating current to the source.

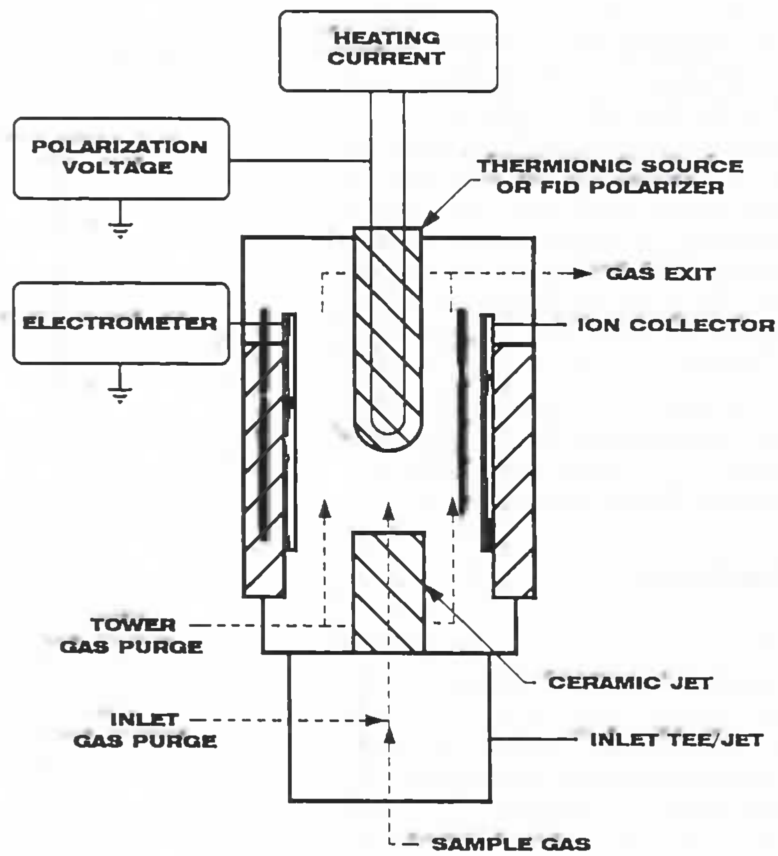
Response:

The FID responds to all organic compounds. In comparison to an FID, the HWCID provides a factor of 2 enhancement for aromatic hydrocarbons relative to alkane hydrocarbons. The HWCID sensitivity is about 100 times less than the FID, but its linearity at high sample concentrations is much better than the FID. By judicious adjustment of the electrical heating of the CFID source, it is possible in the CFID mode to achieve response factors for halogenates which are comparable to hydrocarbons.

TFID: THERMIONIC/FLAME IONIZATION DETECTOR

Equipment:

TFID modules are stand-alone units containing a GC type detector which operates according to the principles of thermionic surface ionization or flame ionization. Each module has a thermally insulated, temperature controlled (50 - 400°C) transducer equipped with a standard Swage type inlet fitting, and supporting gas flow control elements as needed. An air sample pump connected at the module exit port also suffices for the operating gas for some modes. The detector response is determined by the type of thermionic source/FID probe element used, and the type of gases supplied. Available selective modes of thermionic ionization detection include **TID-1-N_2(Air)**, **TID-3-N_2(Air)**, and **NPD**. Available flame ionization modes include a conventional **FID** and the **CFID**. Power and signal measurement for the transducer in the TFID module is provided by a second **CURRENT SUPPLY/ELECTROMETER** module.



Applications:

TFID modules are intended for use in screening sample gas streams evolved from Head Space Analyzers, Thermal Desorbers, Purge and Trap Instrumentation,Super Critical Fluid Extractors, Thermal Analysis Instruments, and any other sample gas generating equipment. When configured with an air pump, the TFID detects electronegatives like NO_2, HF, Cl_2, and I_2 (TID-1-Air mode), or acrylonitrile and methylamine (NPD) in air at ppb levels. The TFID is also a self-contained auxiliary detector system that can be coupled into GCs via heated transfer lines.

CHEMICAL DETECTION by DET

featuring novel applications of the principles of THERMIONIC SURFACE IONIZATION and FLAME IONIZATION

Selective Detection for GC
NPD - BEST N DETECTIVITY
(less than 70 femtograms N/sec)

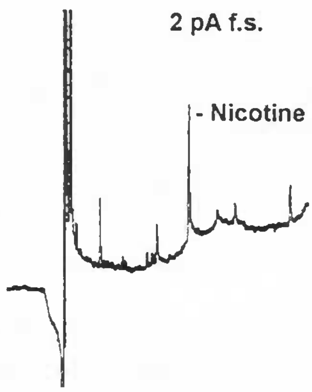
The combination of an Agilent 6890 NPD and a DET TID-4 ceramic ion source (bead) provides state-of-the-art N-selectivity for trace detection of drugs of abuse, pesticides, explosives, and pollutants.

The 6890 NPD hardware features a concentric cylinder ion source - collector electrode geometry for stream-lined gas flow and efficient ion collection. Similar DET equipment is available for HP5890, Varian 3400-3800, and SRI 8610 GC models.

600 femtograms Nicotine

TID-4 in 6890 NPD

2 pA f.s.

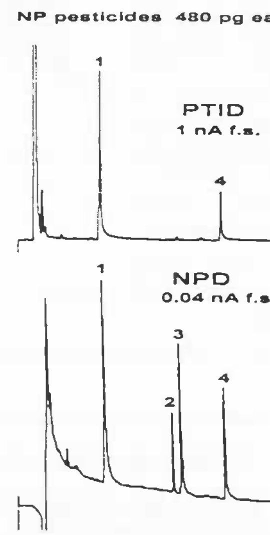


Selective Detection for GC
PHOSPHORUS COMPOUNDS
Very Big Signals with a New PTID

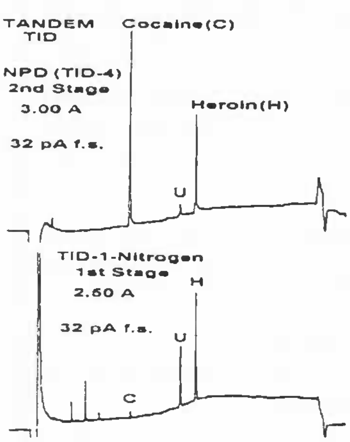
Pesticide Sample:
1-Mevinphos (P)
2-Trifluralin (N)
3-Simazine (N)
4-Methyl Parathion (NP)

NPD detects both P and N. PTID detects only P with signals 10 times bigger than the NPD.

A Phosphorus Thermionic Ionization Detector (PTID) combines surface ionization principles with high flows of Hydrogen and Air for P/C selectivity of 100,000:1, P/N selectivity of 100:1, detectivity of 70 fg P/sec, and a dynamic response range more than 100,000.



Tandem Thermionic Detection for GC
COCAINE - HEROIN



NPD and **TID-1** are two different modes of thermionic ionization.

Ceramic **TID-1** surface operates at 400-600°C in a gas environment of Nitrogen or Air. **TID-1** is non-destructive so it can be combined in series with another detector like the **NPD**.

Ceramic **NPD** surface operates at 600-800°C in an ignited, dilute mix of Hydrogen in Air.

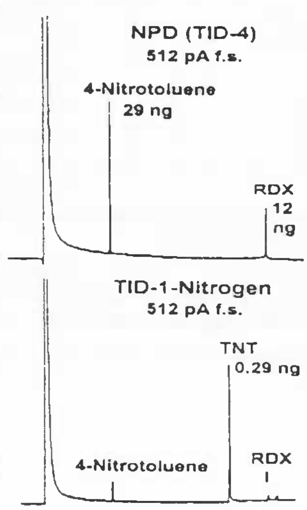
Sample analyzed: NPD detects both Cocaine (C) and Heroin (H). TID-1 detects Heroin and Heroin impurity (U). Tandem combination gives simultaneous TID-1 and NPD signals for each sample injection.

- Femtogram GC Detection -
NITRO-COMPOUNDS like TNT, 2,4-Dinitrotoluene, DNPH-Aldehydes, Methyl Parathion, 4-Nitrophenol, etc.

Unique **TID-1** surface ionization provides better selectivity than ECD and NPD, and needs only Air or N_2 as the detector gas with no requirement for high purity.

TID-1 detection is an inexpensive modification of Agilent 6890 NPD equipment. DET NPD/TID-1 equipment is also available to fit HP 5890, Varian 3400-3800, and SRI 8610 GC models.

EXPLOSIVES Sample: NPD has a big response to RDX and 4-Nitrotoluene. TID-1 has a much larger response to TNT.



DET *innovations in chemical detection*

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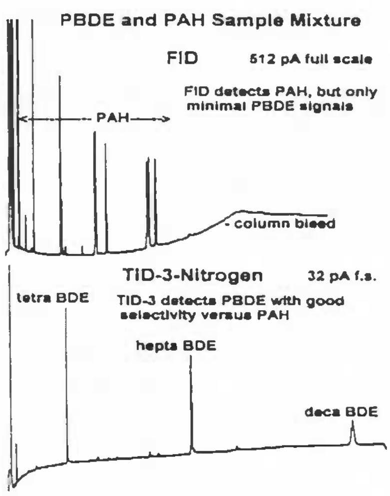
Br Selective Detection for GC
PBDE
(Poly Brominated Diphenyl Ethers)

Negative ionization on a ceramic TID-3 surface detects PBDEs with good selectivity vs. Hydrocarbons.

PBDEs have been used for many years as flame retardants on plastic, foam, and textile products. Recent discoveries that these ubiquitous toxic chemicals are accumulating in environmental and biological media have led to restrictions and bans on their use in many countries around the globe.

Sample analyzed: 18 ng each of 3 PBDEs mixed with 92 ng each of 13 PAH hydrocarbons.



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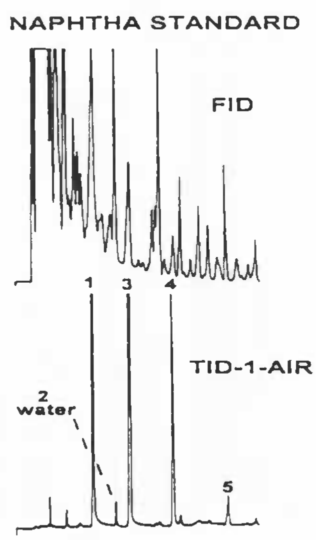
Walnut Creek, CA 94598 USA
Ph 925-937-4203 Fx: 925-937-7581
www.det-gc.com

selective detection for GC
Trace WATER in Solvents and Petroleum Samples

Water is a ubiquitous component of many GC samples. Thermionic ionization on a ceramic TID-1 surface provides a unique combination of sensitivity to Water and selectivity relative to interferences from most Hydrocarbons. Detection of residual Water in organic solvents and complex Petroleum matrices like Gasoline or Diesel fuel is greatly enhanced by this selectivity. TID-1 detection of Water requires only Air as the detector gas, and the Air does not need to be an ultra-high purity grade.



DETector Engineering & Technology

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Walnut Creek, CA 94598 USA
ph 925-937-4203, fx 925-937-7581

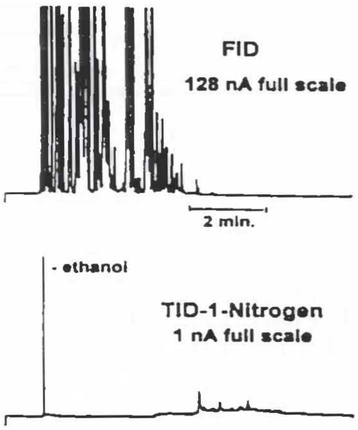
www.det-gc.com

TID-1 and TID-3 modes of detection are simple, inexpensive modifications of Agilent 6890 or DET NPD equipment. Both modes can operate with a detector gas environment of either Nitrogen or Air (no Hydrogen required), with the choice of N_2 or Air depending on the selectivity required. The ceramic coated TID-1 ionization surface is electrically heated to operating temperatures in the range of 400 - 600°C, while the ceramic TID-3 surface operates at 600 - 800°C. Both ion source types are polarized at - 45 Volts with respect to a surrounding ion collector. Ion source polarization and heating power are provided by a stand-alone DET Current Supply module. TID-1 and TID-3 ion source configurations fit into the Agilent 6890 NPD equipment and into DET NPD equipment designed to mount onto HP 5890, SRI 8610, or Varian GCs. Negative ion detection signals can be measured with the Agilent 6890 NPD electrometer, Varian TSD electrometer, SRI FID/NPD electrometer, or with a DET electrometer.

Oxygenate Selective Detection for GC
ETHANOL in GASOLINE

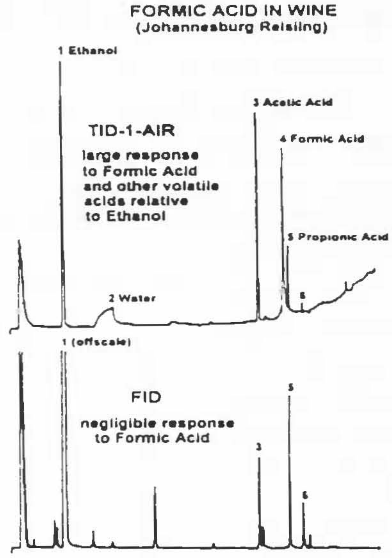
Negative ionization on a ceramic TID-1 surface detects Oxygenates with good selectivity vs. Hydrocarbons.

TID-1 detection provides a simple analysis for the Ethanol additive in gasoline. Only a single gas supply (Nitrogen) suffices for both GC carrier and detector gases. Short analysis times can be used because Ethanol is easily detected amidst many overlapping Hydrocarbon components. TID-1 also detects **Phenols, Glycols,** and other Oxygenated compounds.



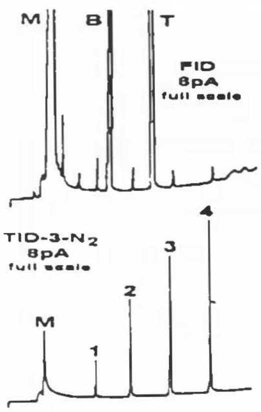
Oxygenate Selective Detection for GC
CARBOXYLIC ACIDS

TID-1 surface ionization with an Air detector gas gives big signals for **Carboxylic Acids** relative to other Oxygenates like Alcohols. TID-1 detection includes **Formic Acid** which is not detected by an FID. TID-1 detection is also **non-destructive** so component aromas can be sensed at the detector exit. H_2O is also detectable to ppm levels



Selective Detection for GC
TRIHALOMETHANES

TID-3 surface catalyzed negative ionization process

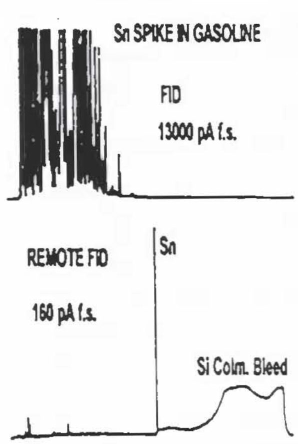


Volatile HALOGENATES detected with a sensitivity of 1 pg/sec, selectivity of 100,000:1 vs. hydrocarbons, and linear response exceeding a range of 10,000 in sample weight.

Unlike other halogen detectors, TID-3 response to Br is significantly more than Cl. Detector gas may be Nitrogen or Air with no requirement for ultra high purity. This detector is much easier and less costly to operate and maintain than an Electrolytic Conductivity Detector.

Sample analyzed:
640 pg each: 1=$CHCl_3$ 2=$CHCl_2Br$ 3=$CHClBr_2$ 4=$CHBr_3$
47,000 pg each: B=benzene T=toluene
2,500,000 pg: M=methanol Solvent: water

Pb – Sn – P – Si
(Lead, Tin, Phosphorus, Silicon)
selective detection with a DET innovation
Organically-Fueled Remote FID

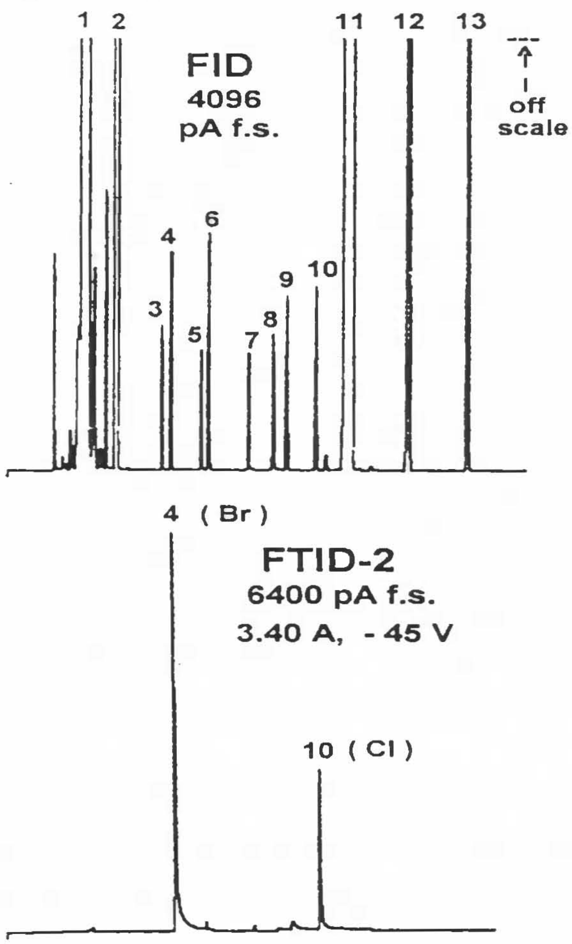


A polarizer and ion collector located several centimeters downstream of a flame jet detect long-lived ion species that originate in a flame fueled by H_2 - CH_4 - Air. Ionization from Hydrocarbon combustion at the jet dissipates before reaching the downstream collector.

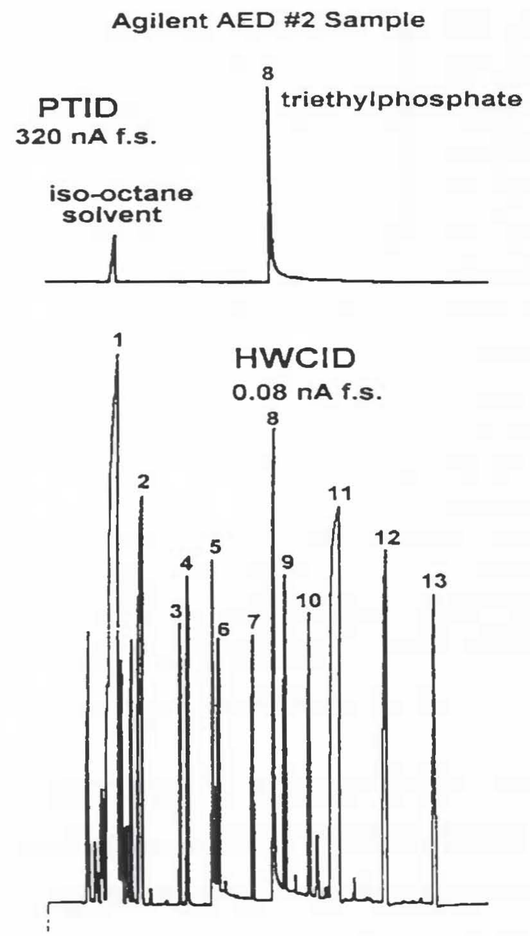
Detectivity of 1 pg/sec for Pb, Sn, P with a selectivity of 500,000:1 versus Carbon.

Sample:
12 ppm tetrabutyltin in gasoline

FTID-2
(Flame Thermionic Ionization)
Halogen Selective



PTID
(Phosphorus Thermionic Ionization)
Phosphorus Selective



Agilent AED test sample #2 components: 1=iso-octane solvent; 2=4.04% n–octane; 3=0.07% 4-fluoroanisole; 4=0.07% 1-bromohexane; 5=0.05% tetraethyl orthosilicate; 6=0.05% n–decane (perdeuterated); 7=0.07% nitrobenzene; 8=0.06% triethyl phosphate; 9=0.05% tert-butyl disulfide; 10=0.08% 1,2,4-trichlorobenzene; 11=4.3% n–dodecane; 12=0.43% n–tridecane; 13=0.13% n-tetradecane.

FTID Principle of Detection: Samples combusted in a H_2-CH_4-Air flame. Electronegative combustion products re-ionized by thermionic surface ionization downstream of the flame.

PTID Principle of Detection: Samples decompose in an ignited H_2-Air chemical boundary layer around a hot thermionic surface. High H_2 and Air flows suppress N response and provide very large P signals.

DET
innovations in chemical detection

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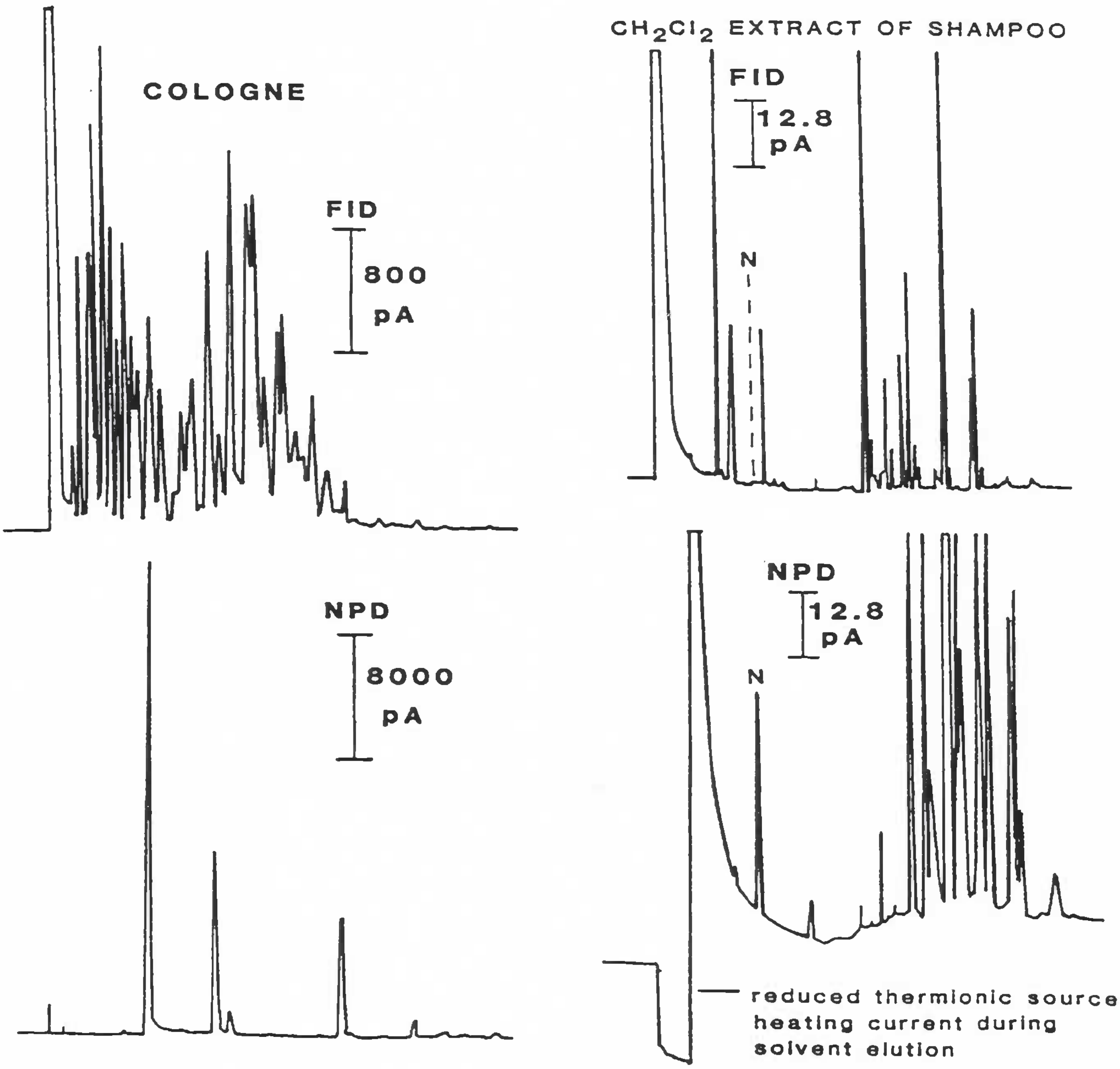
NITROGEN-PHOSPHORUS SELECTIVE DETECTION from DET



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.