

# CERAMIC ION SOURCES FOR NPD AND OTHER MODES

### **1 JANUARY 2016 PRICES**

New Ion Sources (010-9XX-00) \$495 USD each

Recycled Ion Sources (010-9XX-RC) \$460 USD each

(Recycled - Twinex electrical connector & Aluminum connector holder re-used all other parts new - same performance as new source)

XX in part number designates ceramic coating type (e.g., XX = 02 is TID-2, XX = 04 is TID-4, XX = 10 is TID-10, etc.)

### DET ceramic coated Ion Sources are lowest cost replacements for Agilent 6890/7890 and Thermo Trace 1300 NPDs

Example Price Comparison:

DET TID-2 (sharp P peaks) or TID-4 (best N response)	\$495. ea
Agilent Black Ceramic Part 5183-2007 (TID-2 from DET)	\$614. ea
Agilent "Blos" Glass Bead	\$795. ea

Other similar priced DET Ion Sources (eg., TID-10) convert Agilent or Thermo NPD equipment to selective detection of Oxygenates, Halogenates, Nitro compounds, Pyrrole functional groups, compounds with chains of Methylene groups, etc.

Inexpensive detector hardware retrofits for Thermo Trace Ultra NPD and Varian TSD equipped GCs accommodates standard DET Ion Sources for improved NP detection and easy adaptability to other selectivity modes.

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### CERAMIC COATED THERMIONIC ION SOURCES by DET Improved NPD Performance - Extended Detection Modes

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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 Oygenates (especially Phenols, Carboxylic Acids, and Glycols), Nitro-compounds, some Halogenates, Pyrrole functional group, and other electronegative functionalities - operates at 400 - 600°C in inert (N<sub>2</sub>) or oxidizing (air, O<sub>2</sub>) 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 (CH<sub>2</sub>) 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-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 -  $800^{\circ}$ C in a dilute H<sub>2</sub> 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 ( $N_2$ ) or oxidizing (air,  $O_2$ ) gas environments - low picogram detection for Trihalomethanes with minimal peak tailing and greater response for Br versus CI – 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 CI when configured with a stoichiometric mixture of Hydrogen and Air (e.g.,  $H_2 = 5$ , Air = 12.5,  $N_2$  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.,  $H_2 = 20$ , Air = 200,  $N_2$ makeup = 100 mL/min).

**TID-7 (010-907-00)** - Green Ceramic for **Halogenated Pesticides**, **PCBs** - operates in N<sub>2</sub>, Air, or O<sub>2</sub> - 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.

### **DETector Engineering & Technology, inc.**

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### ET CERAMIC ION SOURCES (BEADS) FIT AGILENT 6890/7890 NPD EQUIPMENT





#### FEATURES OF DET ION SOURCES:

Unlike glass NP beads, robust ceramic materials do not soften or melt at the 600 – 800°C temperature required for NP detection;

Wire core protected from corrosive exposure by a unique inert ceramic sub-layer coating;

Unlimited shelf life under ambient conditions;

Wide variety of possible surface coatings for variations in the selectivity of ionization and catalytic activity for converting the same equipment to other selective detection modes.

#### **2 ION SOURCE COATING CHOICES ARE AVAILABLE FOR NP DETECTION:**

TID-2 type (black ceramic) for non-tailing Phosphorus peaks, but Nitrogen response not as large as TID-4 type – recommended for applications requiring P or both N and P detection (e.g., pesticides) - 70fg P/sec detectivity;

TID-4 type (white ceramic) for best possible Nitrogen response, but some tailing of Phosphorus peaks – recommended for applications requiring only N detection (e.g., drugs of abuse) – 70fg N/sec detectivity;

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

Agilent NPD equipment is easily converted to other modes by changing the ion source type and the composition of detector gases – other modes work even better when a stand-alone DET Current Supply is used instead of Agilent's Bead Voltage to power the ion source.

TID Modes – detector gases are Nitrogen, Air, Oxygen, or combinations thereof – selectivity for Nitro compounds, Oxygenates, Halogenates, Pyrrole functional group, etc., depending on the type of ion source used;

CCID Mode – detector gases are Air or Oxygen – selectivity for compounds containing chains of Methylene (CH<sub>2</sub>) functional groups such as Petroleum Hydrocarbons, FAMEs, and Triglycerides, with discrimination between compounds having saturated vs. unsaturated Carbon bonds depending on the concentration of Oxygen in the detector gas.

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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 had 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 GC MODELS (part 010-860-20, price \$2100). Replaces the Varian TSD/NPD hardware which had a side mounted NP bead, with a more optimum concentric cylinder geometry and a top mounted ion source. DET tower structure is about half the size of the Varian tower. 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/ jet. Any DET ion source priced at \$495 each can be accommodated. Hardware is compatible with Varian TSD electronics that suffice for NPD. However, 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. 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.

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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

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Tower body is 0.75 inch stainless steel hexagonal stock. Base of tower can be fit with a Swage or Tube gas fitting, or machined to custom fit existing detector base on a GC. Fiberglass insulated ion source cable is available with a standard Twinex connector, or with no connector. Standard signal probe arm terminates in a BNC connector and extends 4.75 inches from side of the tower body. If needed, center of probe arm can be bent 90 degrees to reduce extension to 2.50 inches from tower. Gas flows in through bottom of tower and out the back side.



Varian/Bruker GCs – DET tower base and flange designed to custom fit existing FID/TSD base.



Thermo Trace Ultra GC – custom fits Thermo base. Signal probe terminates in an SMA type connector.



SRI Instruments GC – DET tower attaches to 1/8 inch diameter jet via a Swage fitting. 1/16 inch side Swage port adds Air flow to detector. Custom signal probe arm extends 1.5 inches from tower and bends down in unshielded bare wire.

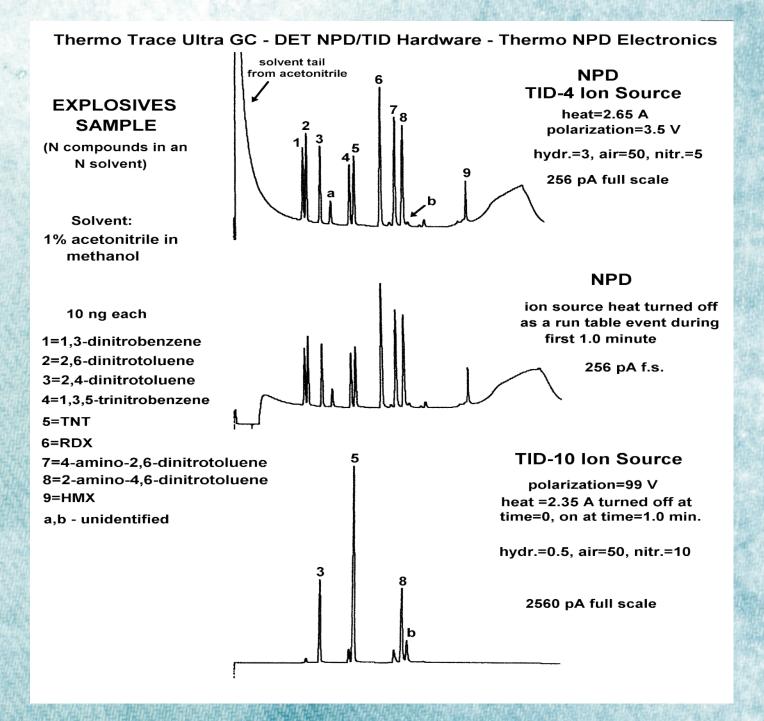
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Comparison of NPD vs. TID-10 modes of selective detection - same hardware structure and electronics with different type ion sources and different type detector gases. NPD detected all the explosive compounds, while TID-10 provided high selectivity and very large responses to compounds 3, 5, and 8 which contained a Nitro (NO<sub>2</sub>) 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.

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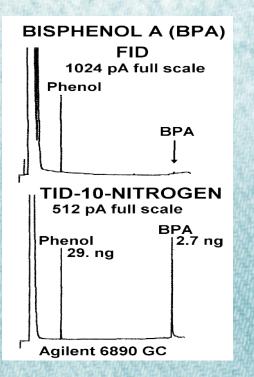
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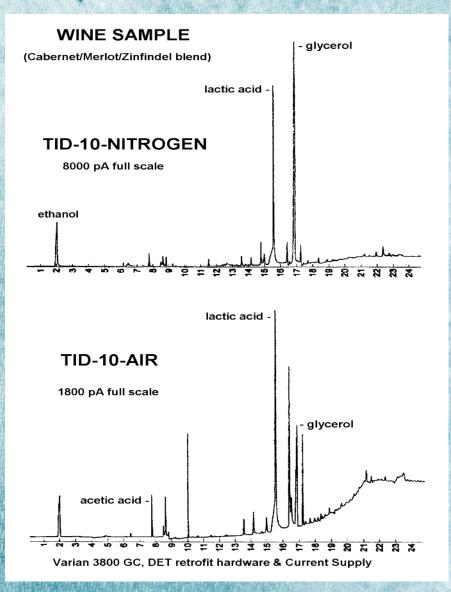
### TID-10 ION SOURCE

inexpensive conversion of NPD equipment to selectivity for Oxygenates greatly expands applications for the equipment (requires only Nitrogen or Air as detector gas)



Phenols, Carboxylic Acids, and Glycols produce especially large TID-10 responses compared to other Oxygenate classes.

Agilent NPD equipment with TID-10 ion source and Nitrogen supplied through all 3 detector gas lines. Stand-alone DET Current Supply used instead of Agilent Bead Voltage in order to polarize ion source at - 45 V for 10 times better signal-to-noise. Agilent Bead Voltage suffices for similar selectivity, but has lower polarization and signal magnitudes.



TID-10 selectivity can be different depending on whether the detector gas is Nitrogen, Air, or a combination thereof. Existing Varian TSD electronics suffice for selectivity, but lower ion source polarization voltage provides smaller signal-to-noise than a stand-alone DET Current Supply at - 45 V.

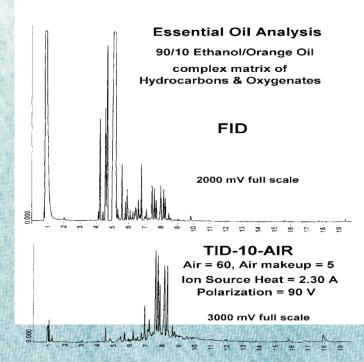
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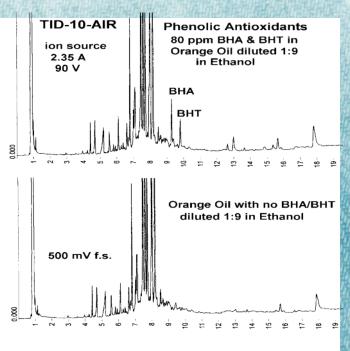
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#### THERMIONIC SURFACE IONIZATION - SELECTIVE DETECTION OF OXYGENATES





#### **ESSENTIAL OILS**

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

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

Data were generated using a Thermo Trace Ultra GC retrofitted with DET NPD/TID hardware mounted on Thermo's NPD detector base, and operated using Thermo's NPD electronics.

Air was supplied to the detector through the 3 gas lines that normally provide "H<sub>2</sub>", Air, and Makeup gases to an NPD.

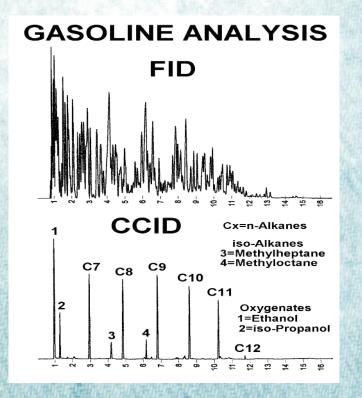
BHA and BHT are Phenolic Antioxidants that are widely used as preservatives in many food products. TID-10-Air detection provides enhanced responses for Phenol type compounds relative to other type constituents of Essential Oils.

Similar data can be achieved with simple adaptation of existing NPD equipment on the Thermo Trace 1300 GC. Agilent NPD equipment also suffices, although best detectivity is achieved by a stand-alone DET Current Supply substituted for Agilent's Bead Voltage as the power supply for the ion source. For Varian and SRI Instruments NPD GC models. 0 selectivity uses inexpensive DET Retrofit hardware with the existing NPD electronics, or optimally with a **DET Current Supply.** 

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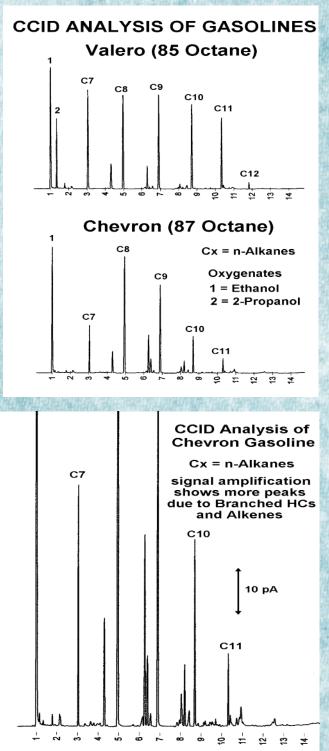
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Catalytic Combustion Ionization (CCID) is a unique means of detecting n-Paraffin constituents in an otherwise complex petroleum matrix such as Gasoline. Other constituents such as Olefins and iso-Paraffins that contain chains of Methylene ( $CH_2$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

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



Same chromatographic conditions were used for all data.

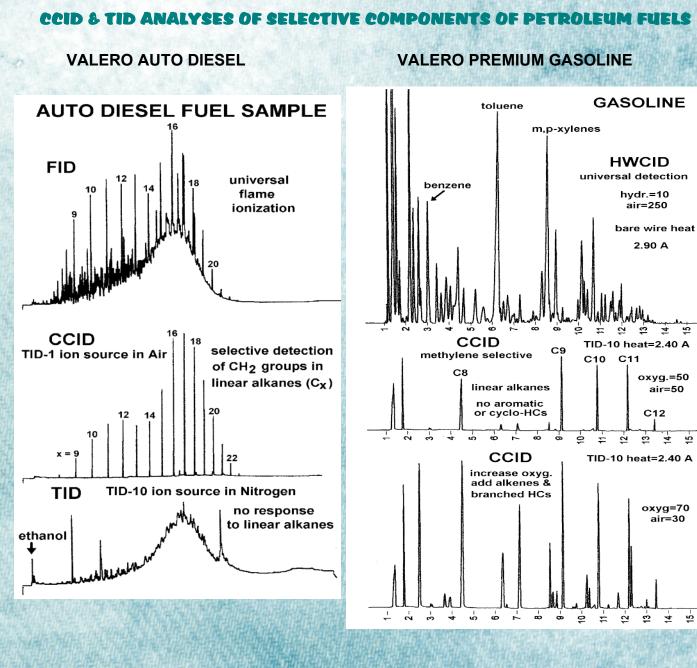
DET retrofit on Thermo Ultra GC, Air=30, O<sub>2</sub> makeup=50mL/min, TID-10 ion source heat=2.45A, polariz.= - 90V. 0.5: L gasoline, 30m x 0.53mm DB1, He=8, 40-60°C at 5°C/min, 60-190°C at 10°C/min, detector=250°C.

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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.

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