DET REPORT

NO. 42 AUGUST 2001

1. WINE ANALYSIS - TID-1 DETECTION OF DIACETYL; ACETIC, FORMIC, PROPIONIC, AND LACTIC ACIDS; GLYCEROL; AND VANILLIN.

2. DIESEL FUEL ANALYSIS - TID-1 AND NPD DETECTION OF CARBAZOLES, OXYGENATES, AND POLYNUCLEAR HYDROCARBONS.

3. NPD & TID-1 ON "GASLESS" SRI GC - EXPLOSIVES, PESTICIDES, DRUGS.

By P .L. Patterson

1. WINE ANALYSIS - TID-1 DETECTION OF DIACETYL; ACETIC, FORMIC, PROPIONIC, AND LACTIC ACIDS; GLYCEROL; AND VANILLIN.

The selective detection of Oxygenates by TID-1 thermionic surface ionization has been described previously in numerous DET Reports. Unlike the Flame Ionization Detector which is often used to detect Oxygenates as well as Hydrocarbons, the TID-1 detector does not require either Hydrogen or a flame. Rather, TID-1 ionization works with either an inert detector gas environment like Nitrogen, or an oxygen containing environment like Air or O_2 , and these do not need to be ultra-high purity grade gases. From past work, we know that most Oxygenates are detected with the largest signals and the best selectivity versus Hydrocarbons when a Nitrogen environment is used. When Oxygen is introduced into the detector, TID-1 response to compounds like Alcohols, Ketones, and Aldehydes are reduced significantly, and responses to the CH_2 functional group in high concentrations of linear chain Hydrocarbons begin to appear. However, TID-1 responses for compounds like Phenols and Carboxylic Acids remain very much larger than other Oxygenates irrespective of whether an inert or oxidizing gas environment is used. These characteristics mean that TID-1 ionization can be a very effective means of detecting minor and trace constituents in beverage, fragrance, or food samples where the major constituents are Water and/or Alcohols. Another attractive feature of this detector is that it is non-destructive, so aromas from different components in a sample can be sensed at the detector exit whether or not there is a corresponding TID-1 electrical signal.

This report provides some illustrative applications of TID-1-Air ionization in wine analyses. Some data on TID-1 analyses of wines have been reported in previous DET Reports, but we now have been able to identify a few more of the peaks detected by this unique method of ionization. Figure 1 demonstrates how TID-1-Air ionization enhances responses to Volatile Acids in wine relative to the Ethanol peak.

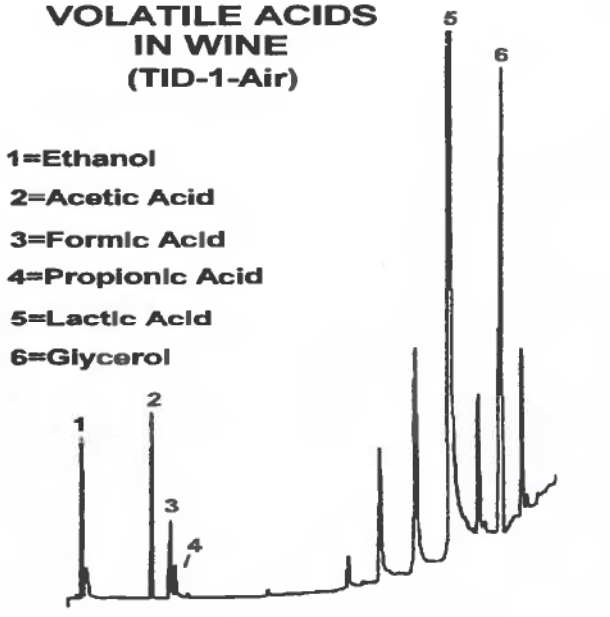

Figure 1. 1.7µL Bordeaux Wine. 30m x 0.53mm x 2µm DBWAXetr, He=15mL/min, 110-240°C at 4°C/min. Injector=240°C, Detector=280°C, Detector Air=60mL/min. TID-1 heat=2.55A, polarization=-45 V. 128pA full scale. Peak 5 was off scale. Varian 3800 GC.

CHROMalytic TECHnology PTY LTD
ABN 14 643 445 058
Website: http://www.chromtech.net.au
PO Box 435, 232 Forest Rd, Boronia, Victoria 3155, Australia
Tel: +61 3 9762 2034 Fax: +61 3 9761 1169 E-mail: sales@chromtech.net.au

Although Ethanol was a dominant constituent of this wine sample, the Air environment in the TID-1 detector effectively suppressed the Ethanol peak, such that TID-1 signals for Lactic Acid, Glycerol, and Acetic Acid each exceeded that of the Ethanol. The wine analyzed in Figure 1 was a 1998 Bordeaux blend of Cabernet Sauvignon, Merlot, and Cabernet Franc. In addition to the labeled peaks, there were obviously several other prominent TID-1 peaks which have not yet been identified.

Figure 2 compares FID and TID-1-Air chromatograms for the same wine sample as Figure 1, and illustrates how each detector responds differently to the constituents in the sample. One objective of the analysis in Figure 2 was to detect the compound Diacetyl because it can contribute to the overall taste and aroma of wine although present in very trace amounts. From sniffing vapors emanating at the TID-1 detector exit, the characteristic "buttery" aroma of Diacetyl (peak 2) was identified right after

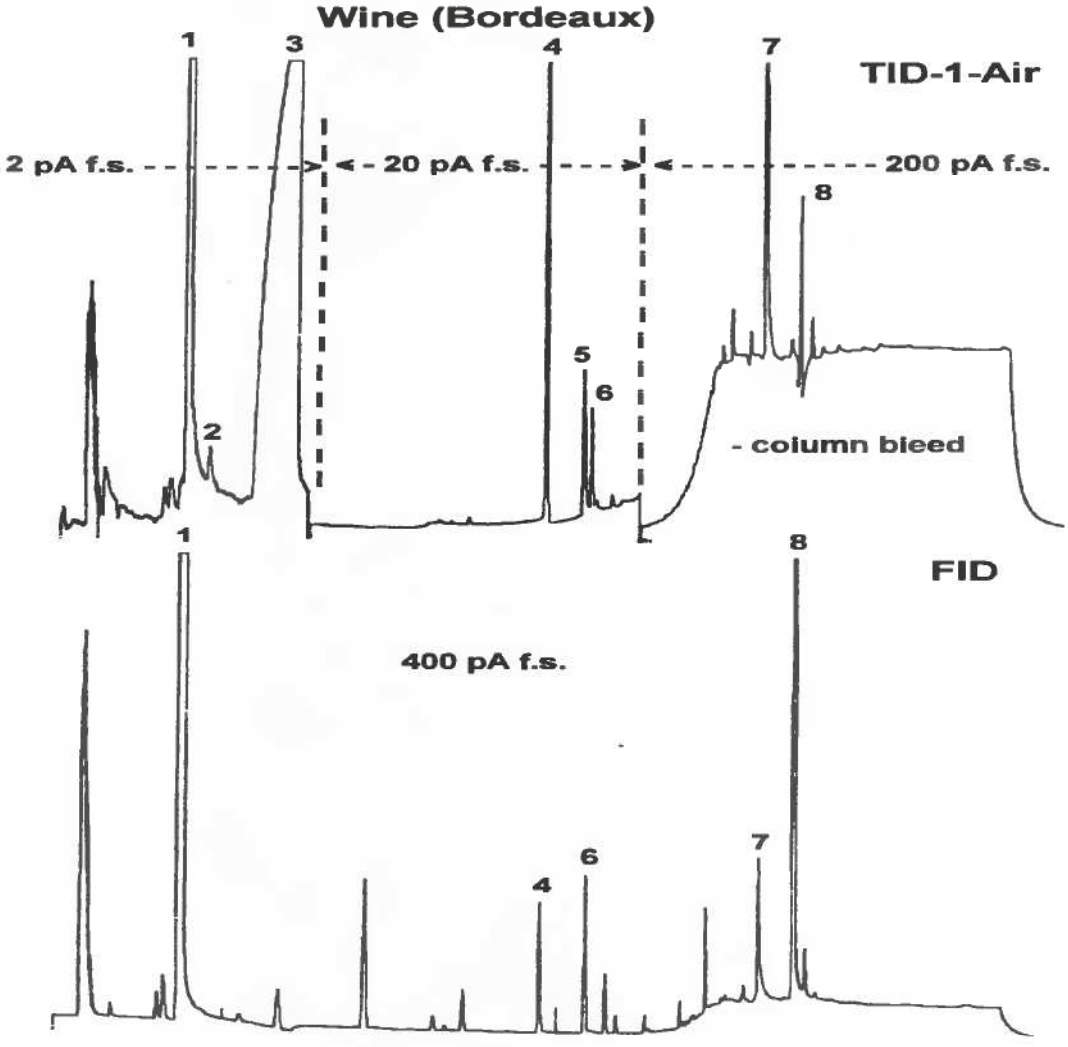

Figure 2. 0.6µL Wine injected. 1=Ethanol, 2=Diacetyl, 3=Water, 4=Acetic Acid, 5=Formic Acid, 6=Propionic Acid, 7= Lactic Acid, 8=Glycerol. Same column as Figure 1. He=8mL/min. 50-90°C at 6°C/min, 90-160°C at 8°C/min, 160-240°C at 40°C/min, 240°C-8min. TID-1 peaks 1, 3, 4, and 7, and FID peaks 1 and 8 were off scale.

CHROMalytic TECHnology PTY LTD
ABN 14 643 445 058
Website: http://www.chromtech.net.au
PO Box 435, 232 Forest Rd, Boronia, Victoria 3155, Australia
Tel: +61 3 9762 2034 Fax: +61 3 9761 1169 E-mail: sales@chromtech.net.au

the elution of Ethanol from the detector. With the GC column that was used, Diacetyl could not be detected with the FID because of the close elution with the large Ethanol peak. However, with the TID-1-Air detector, the Ethanol response was sufficiently suppressed to allow Diacetyl (peak 2) to be detected in that chromatogram.

In Figure 2, Formic Acid (peak 5) and Water (peak 3) were two additional sample components detected by TID-1 ionization but not by the FID. Conversely, there were component peaks in the FID chromatogram that did not appear in the TID-1 chromatogram.

Like Diacetyl, Vanillin is another trace constituent in wine that can contribute a very characteristic aroma and flavor. The TID-1 chromatogram in Figure 3 shows a small peak (10) identified as Vanillin by its unique aroma as well as by its coincidence with the retention time of a Vanillin standard.

Figure 3 also shows a comparison of TID-1-Air, FID, and NPD (TID-4) chromatograms for the Bordeaux wine sample. The NPD revealed some prominent peaks which were not present in either the TID-1 or FID chromatograms. For example, peak 9 was large and peak 8 was very small in the NPD chromatogram, whereas the opposite was true in the TID-1 and FID chromatograms. While both the NPD and FID detectors destroy the sample, the TID-1 detector does not. Hence, even

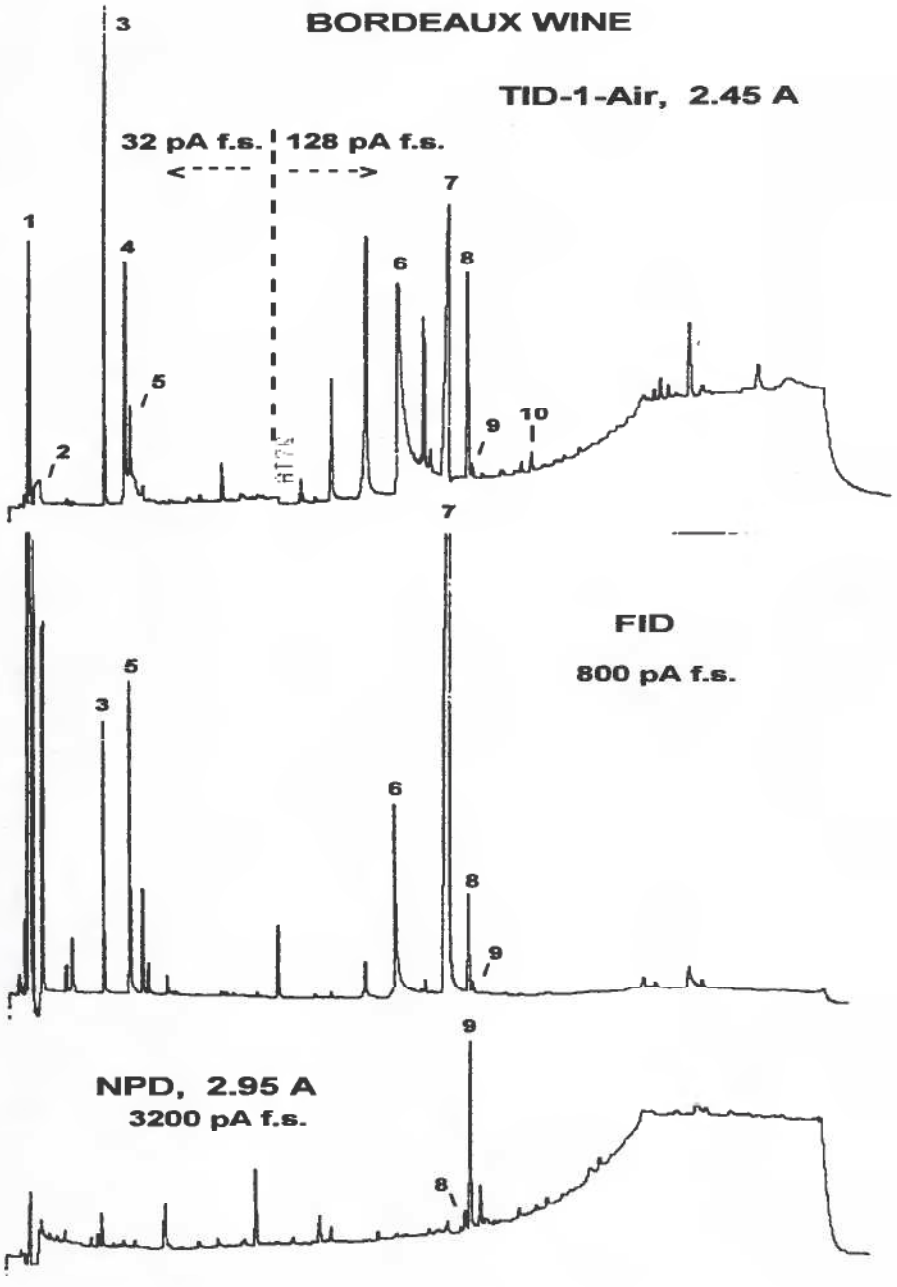

Figure 3. 1.8µL Wine. 1=Ethanol, 2=Water, 3=Acetic Acid, 4=Formic Acid, 5=Propionic Acid, 6=Lactic Acid, 7=Glycerol, 8 & 9=unidentified, 10=Vanillin. 30m x 0.53mm x 1.0µm DB-WAXetr, He=10mL/min, 100-240°C at 5°C/min, 240°C-8min. Hydrogen/Air=20/200 mL/min for FID, =3/60 mL/min for NPD, = 0/60 mL/min for TID-1-Air. TID-1 source = 2.45 A, -45 V; NPD source = 2.95 A, -5V; FID Probe = 3.40 A for flame ignite, 0 A thereafter, -45 V.

though there may not be a TID-1 peak generated, sniffing the TID-1 exit vapors allows characteristic aromas to be associated with different retention times. In some cases, these TID-1 exit aromas can be correlated with the appearance of an FID or NPD peak at that same retention time.

The Lactic Acid peak in the TID-1 chromatogram of Figure 3 tailed considerably on the column that was used. This column had a lighter coating than the column used for Figures 1 and 2. The Lactic Acid tailing got progressively worse with repeated direct injections of the wine sample. For example, the FID chromatogram was generated prior to the TID-1 chromatogram, and the FID peak for Lactic Acid did not tail as badly as the TID-1 peak. This was confirmed to be an effect at the injector end of the column by cutting off the first couple inches of column, and having the tailing disappear for the first few injections thereafter.

The data in Figures 1 - 3 were generated with DET equipment mounted on a Varian 3800 GC. The DET hardware consisted of a TID/FID Tower/Jet Assembly mounted onto an existing Varian FID detector base. The TID-1, NPD, and FID modes of detection were obtained by installing TID-1, TID-4, or FID Probe elements into the DET tower, respectively. TID-1 and TID-4 were ceramic coated Ion Source elements, while the FID Probe was an uncoated loop of wire that served as both flame ignitor and polarizer. The TID/FID elements were powered with a stand-alone DET Current Supply module, and negative ion signals from the detectors were measured with a Varian TSD Electrometer. Hydrogen and Air flows of the appropriate magnitudes for each mode were supplied via Varian pneumatics controls through the two gas lines to the Varian FID base. Our Varian GC had intermittent instability problems with the Electronic Flow Control (EFC) of the detector makeup gas, so we eliminated the need for that by installing the end of the 0.53 mm GC columns to the very top of a DET ceramic-lined jet. Therefore, carrier gas and samples eluted from the GC column directly into the flowing Hydrogen and Air gases of the detector without exposure to the internal volume of the jet. For FID data, Hydrogen and Air flows were 20 and 200 mL/min, respectively. For TID-1 and NPD data, Hydrogen and Air were 3 and 60 mL/min, respectively. A difference between NPD and TID-1 modes was the TID-4 Ion Source of the NPD was powered with a higher heating current in order to ignite the Hydrogen-Air chemistry required for NP selectivity.

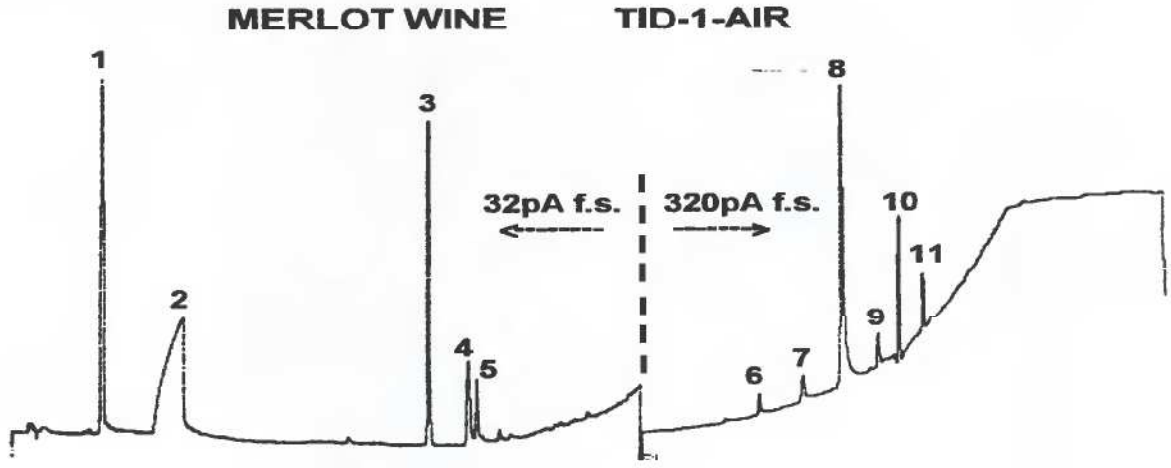

Figure 4. 0.4µL wine, 1=Ethanol, 2=Water, 3=Acetic Acid, 4=Formic Acid, 5=Propionic Acid, 8=Lactic Acid, 10=Glycerol. 30m x 0.53mm x 2µm DB-WAXetr, He=10mL/min, 50-240°C at 6°C/min, 240°C-5 min.

CHROMalytic TECHnology PTY LTD
ABN 14 643 445 058
Website: http://www.chromtech.net.au
PO Box 435, 232 Forest Rd, Boronia, Victoria 3155, Australia
Tel: +61 3 9762 2034 Fax: +61 3 9761 1169 E-mail: sales@chromtech.net.au

Figures 4, 5, and 6 compare TID-1-Air chromatograms for five different varietal wines. Identified peaks in these chromatograms were Ethanol (1), Water (2), Acetic Acid (3), Formic Acid (4), Propionic Acid (5), Lactic Acid (8), and Glycerol (10). Although not yet identified, peak 6 corresponded to the elution of a strong "burned sugar" aroma at the detector exit. We have not yet been able to assign a similar descriptive terminology to the characteristic aromas of the other unidentified peaks. The distribution of peaks as well as relative peak heights differ for the wines analyzed. The three red wines in Figures 4 - 6 all had prominent Acetic Acid and Lactic Acid peaks, while the Johannesburg Reisling had an enhanced Formic Acid peak as well as a large unidentified peak (12) which was not present in the other four chromatograms.

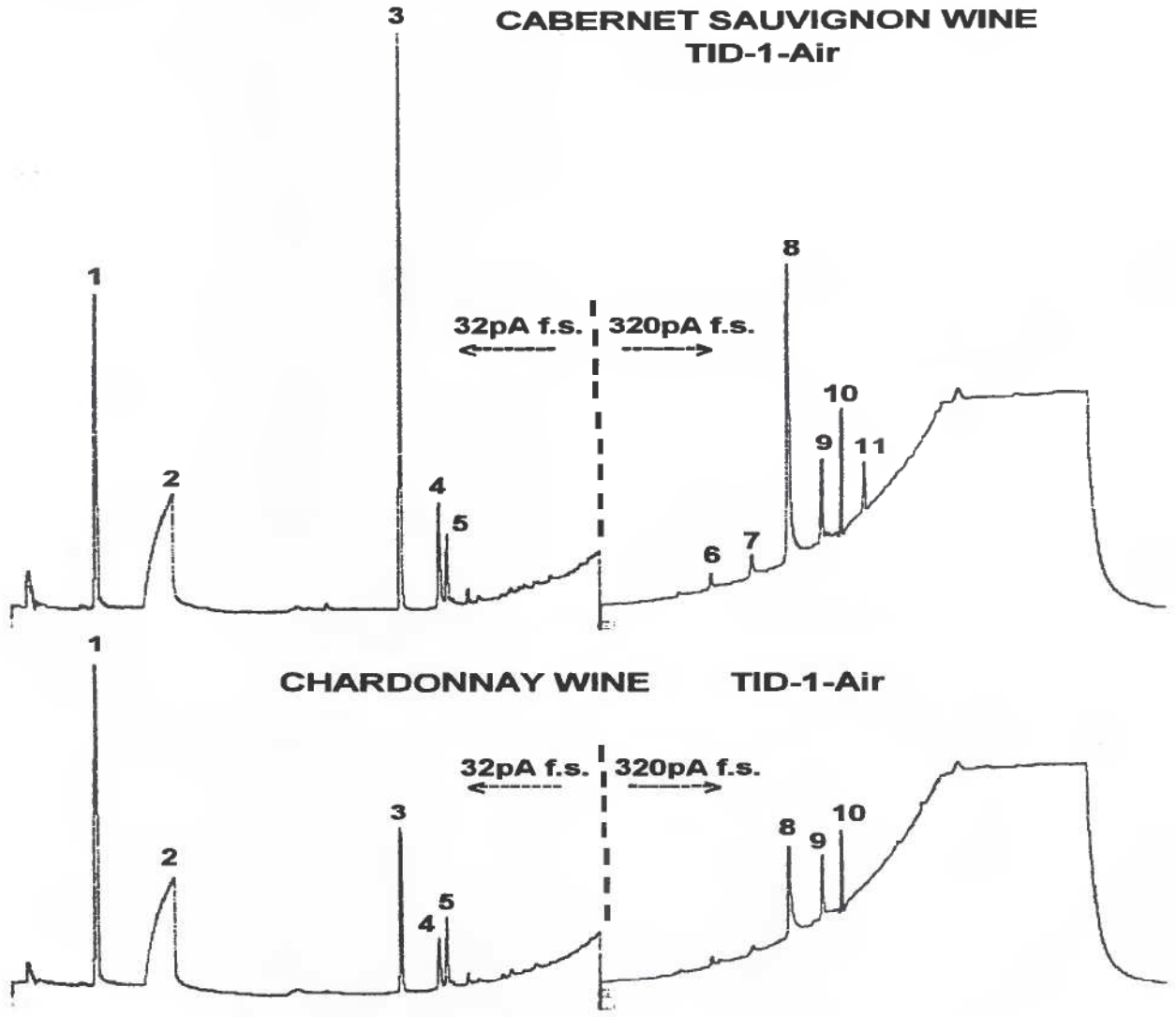

Figure 5. 0.4µL wine, same conditions and peak labels as Figure 4.

ABN 14 643 445 058
Website: http://www.chromtech.net.au
PO Box 435, 232 Forest Rd, Boronia, Victoria 3155, Australia
Tel: +61 3 9762 2034 Fax: +61 3 9761 1169 E-mail: sales@chromtech.net.au

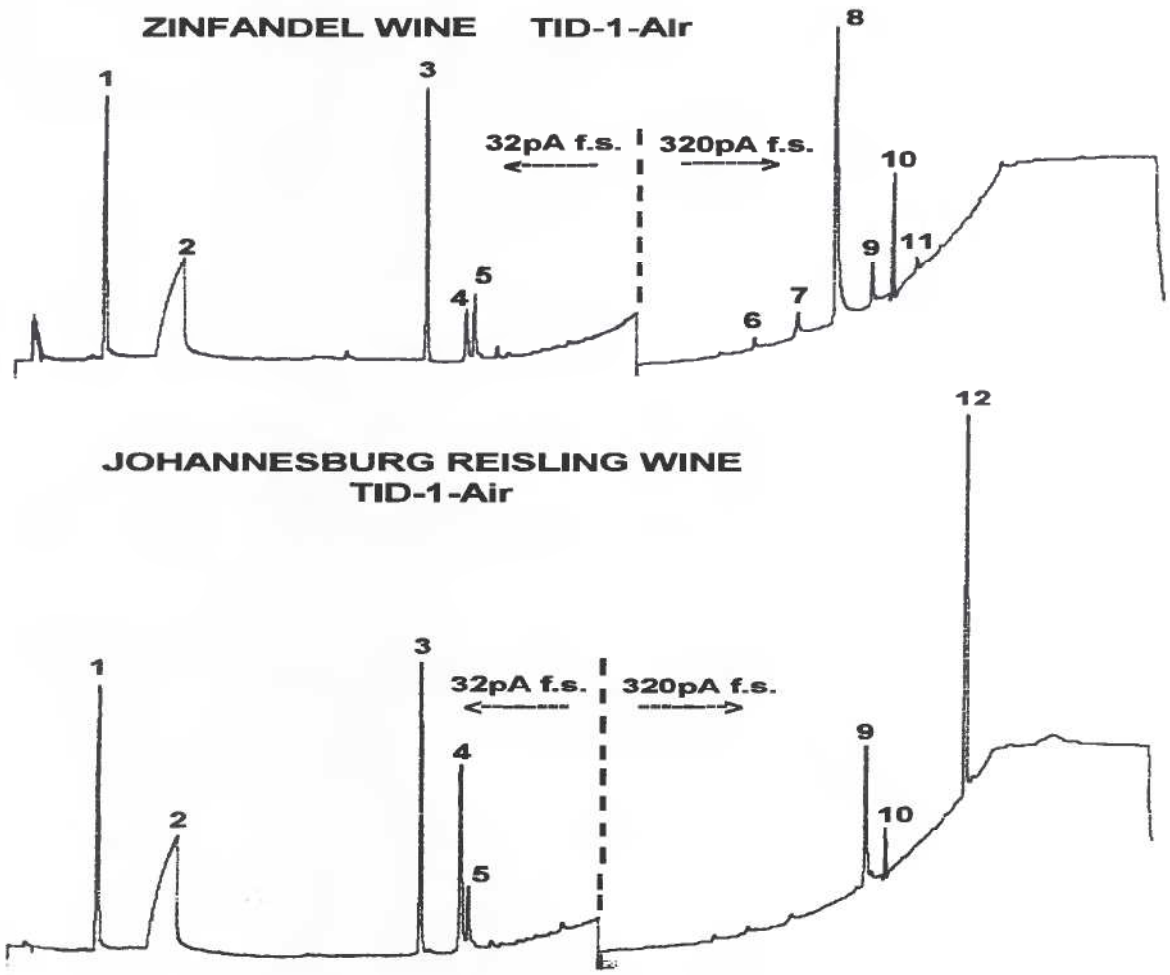

Figure 6. 0.4µL wine, same conditions and peak labeling as Figure 4.

2. DIESEL FUEL ANALYSIS - TID-1 AND NPD DETECTION OF CARBAZOLES, OXYGENATES, AND POLYNUCLEAR HYDROCARBONS.

Thermionic ionization on a TID-1 type ceramic surface exhibits its best selectivity versus Hydrocarbons when that surface is operated in an inert gas environment of Nitrogen. Previous DET Reports have often used analyses of Diesel Fuel samples to demonstrate this TID-1 selectivity. In these analyses, there were typically a significant number of TID-1 peaks occurring at late retention times in the chromatogram, well after the major Hydrocarbon components of the sample had eluted. In this report, we review some of the known characteristics of TID-1-N_2 detection as they may be applied to help identify the late eluting Diesel Fuel peaks.

The TID-1 detection process involves the formation of negative ions when gas phase sample molecules impact a hot, catalytically active, solid surface. Gas phase negative ions formed by extraction of electrons from the surface are subsequently measured at a surrounding collector electrode. Previous work has demonstrated that Oxygenates are among the classes of heteroatom compounds that are selectively ionized, and among the

CHROMalytic TECHnology PTY LTD
ABN 14 643 445 058
Website: http://www.chromtech.net.au
PO Box 435, 232 Forest Rd, Boronia, Victoria 3155, Australia
Tel: +61 3 9762 2034 Fax: +61 3 9761 1169 E-mail: sales@chromtech.net.au

Oxygenates, Phenols are especially well detected. This is demonstrated in the data of Figures 7 and 8. Figure 7 shows a comparison of FID and TID-1-N_2 chromatograms for a mixture of singly-substituted benzene compounds, where the substituents were electronegative atoms and functional groups. From the TID-1 chromatogram, it can be determined that the selectivity of the Phenol OH peak response per gram of substance was at least greater than 10,000:1 versus the F component; at least greater than 1000:1 versus the Cl and Br components; 85:1 versus the NO_2 component; and 45:1 versus the NH_2 component.

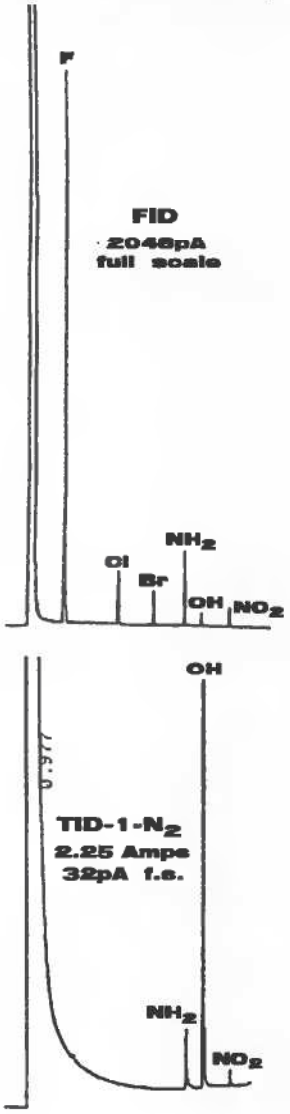

Figure 7. Sample: F=Fluorobenzene(760ng), Cl=Chlorobenzene(76ng), Br=Bromobenzene(76ng), NH_2=Aniline(76ng), OH=Phenol(11ng), NO_2=Nitrobenzene(34ng) in Methanol. 30m x .53mm DB624, He=8mL/min, 70-180°C at 10°C/min.

Figure 8 compares FID and TID-1-N_2 responses for a mixture of Phenols, Alcohols, and Aromatic Hydrocarbons. Both FID and TID-1 chromatograms are displayed at the same full scale sensitivity to illustrate that the TID-1 mode was unresponsive to the Hydrocarbons, and had greater sensitivity than the FID for the Phenols. The TID-1 chromatogram demonstrates that this detection mode also provides selectivity for Alcohols versus Hydrocarbons, although at a substantially lower absolute sensitivity than for the Phenols.

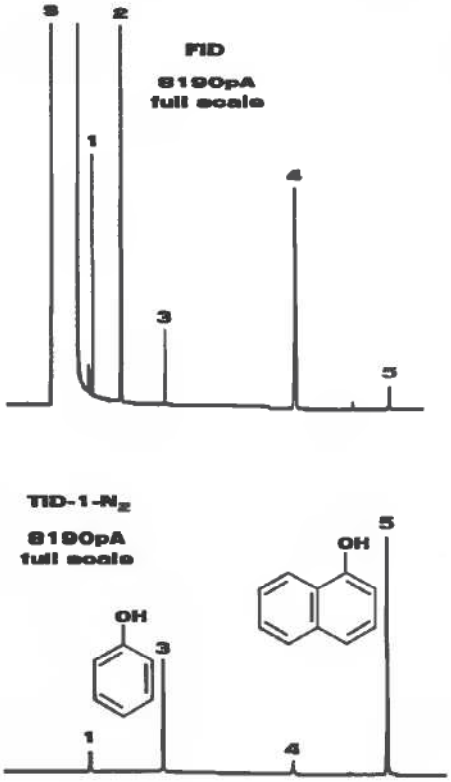

Figure 8. S=Benzene solvent, 1=260ppm Cyclopentanol, 2=990ppm p-Xylene,3=51ppm Phenol, 4=350ppm n-Decanol, 5=51ppm 1-Naphthol. 30m x .32mm HP-5,He=3 mL/min, 35-175°C at 10°C/min.

Figures 9 and 10 illustrate TID-1 responses to Polynuclear Nitrogen and Hydrocarbon compounds. Figure 9 compares FID, NPD, and TID-1-O_2 responses for a mixture of 320 ng each of Indene, Naphthalene, and Fluorene; 66ng each of Indole and Carbazole; and 78 ng of Quinoline in a Toluene solvent. Compared to the FID, the NPD provided both selectivity and sensitivity enhancement for all

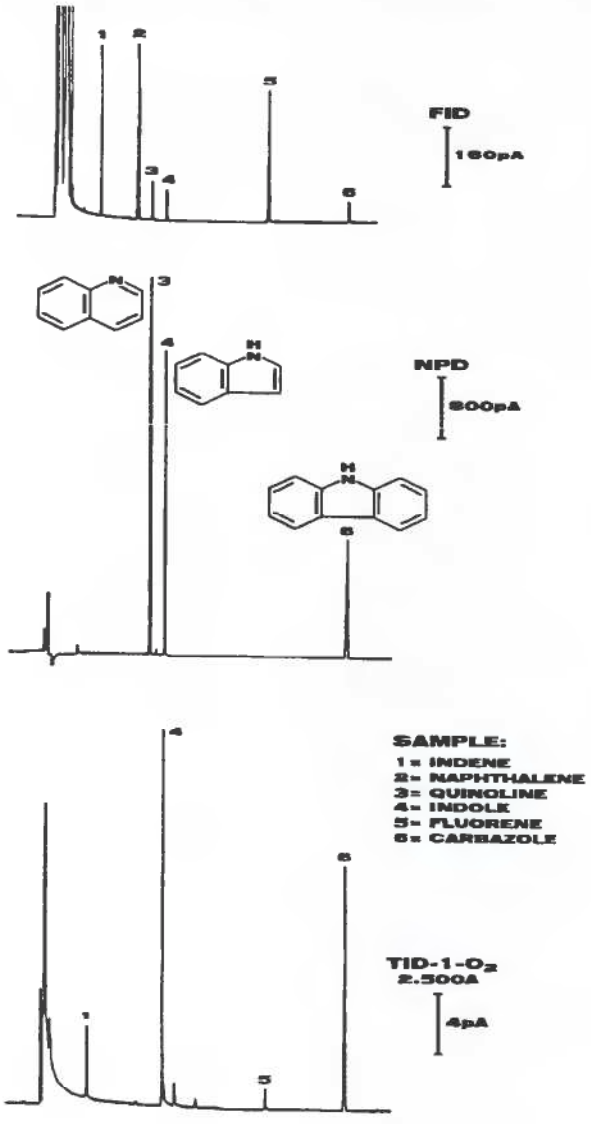

Figure 9. Sample: 1=320ng Indene, 2=320ng Naphthalene, 3=78ng Quinoline, 4=66ng Indole, 5=320ng Fluorene, 6=66ng Carbazole. 30m x .53mm DB-5ms, He=10mL/min, 70-190°C at 8°C/min.

the N-compounds in the sample. In contrast, the TID-1 mode provided selectivity for the Pyrrole Heterocycles, Indole and Carbazole, but no response for the Pyridine Heterocycle, Quinoline. TID-1 also exhibited some selective response to Indene and Fluorene, the Hydrocarbon analogs of Indole and Carbazole. Although the TID-1 gas environment was O_2 for the data in Figure 9, an N_2 environment provides similar conclusions regarding relative peak responses. The selective detection of Fluorene versus other PolyAromatic Hydrocarbons is further demonstrated in Figure 10.

Figure 11 compares FID, NPD and TID-1-Nitrogen chromatograms for a sample of Diesel Fuel. At the sensitivity displayed for the FID, many of the dominant n-Alkane peaks were off scale. Both the NPD and TID-1 chromatograms exhibited a number of peaks at late retention times after most of the Hydrocarbons had eluted. The peak labeled "C" in

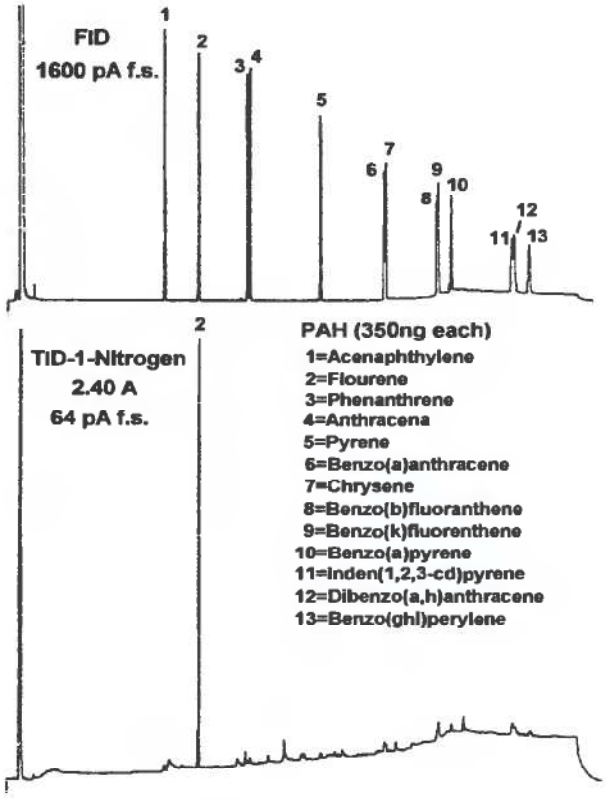

Figure 10. PAH in Acetone solvent. 30m x .53mm DB5ms, He=10mL/min, 100-300°C at 8°C/min, 300°C-7min.

both the NPD and TID-1 chromatograms corresponds to the retention time of Carbazole. The Nitrogen compounds indicated by the "N" bracket in the NPD chromatogram appeared also in the TID-1 chromatogram in the same pattern of retention times but with differing relative peak heights. From the observations of Figure 9, it can be concluded that the N-compounds in this Diesel sample were not Pyridine-type Heterocycles, and were most likely Pyrrole-type Carbazoles. In the TID-1 chromatogram, the peaks indicated by the "O" bracket were not N-compounds since they did not appear in the NPD chromatogram. Rather they were most likely Oxygenates such as Phenolic compounds, and/or Polynuclear Hydrocarbons like Fluorene which contain a five member functional group within their molecular structure. The peak labeled "F" in the TID-1 chromatogram corresponds to the retention time of Fluorene. There were no TID-1 peaks corresponding to the retention times of Indene, Indole, or 1-Naphthol.

When comparing different types of ionization detectors, the absolute signal magnitudes measured in picoAmps provides an indication of their relative efficiencies for ionizing sample molecules. In Figures 9 and 11, note that the NPD not only provides selectivity for N-compounds, but it also provides greater ionization efficiency for those compounds when compared to an FID. The N-compounds are also non-volatile constituents of Diesel fuel samples. Hence, in applications such as identifying petroleum spills, an NPD fingerprint of a weathered Diesel fuel is quite similar to the NPD fingerprint of an unweathered Diesel sample.

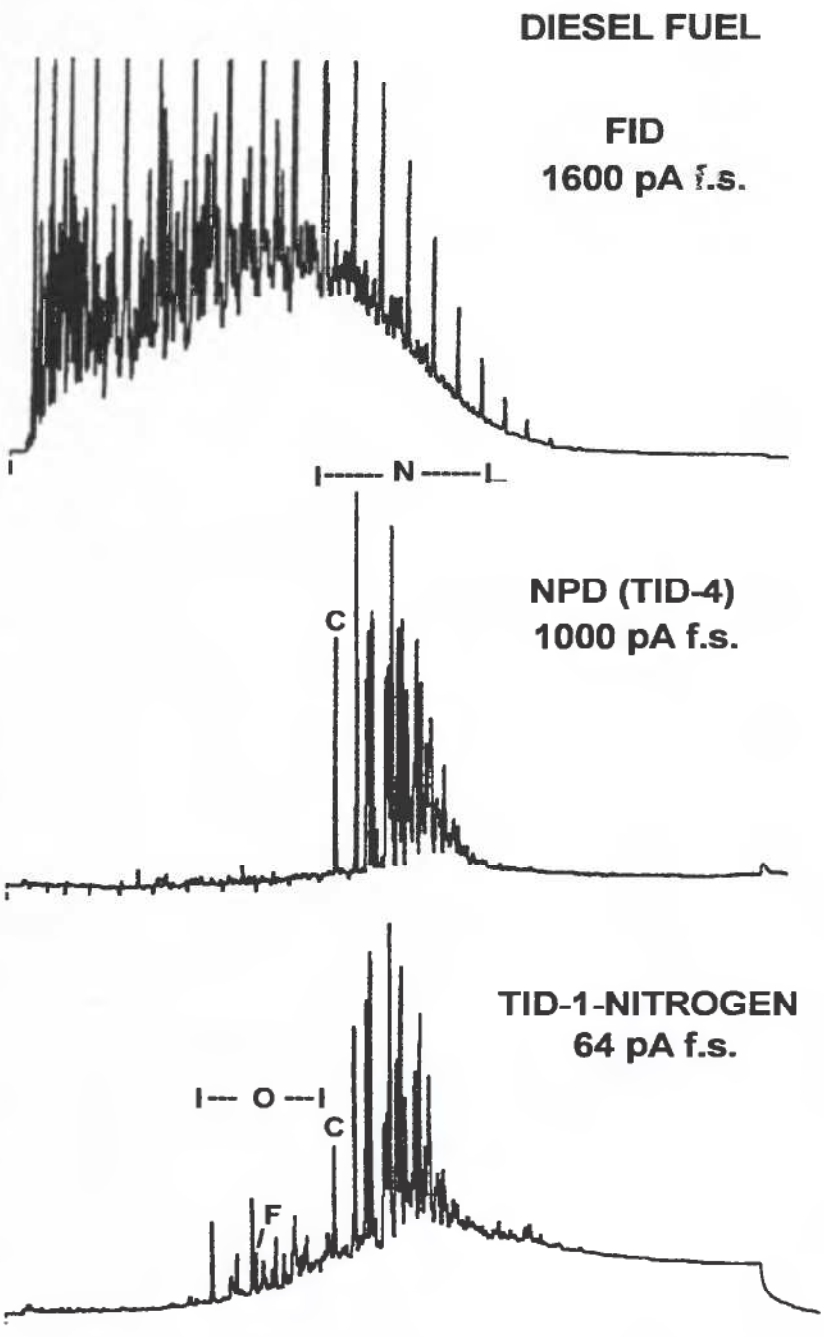

Figure 11. 0.1µL Diesel Fuel injected. 30m x .53mm DB5ms, H=10mL/min, 100-280°C at 8°C/min, 280°C-10min.

DET REPORT

NO. 42 AUGUST 2001

3. NPD & TID-1 ON "GASLESS" SRI GC - EXPLOSIVES, PESTICIDES, DRUGS.

DET Report #41 (June 2001) described DET Thermionic/Flame Ionization detector hardware and electronics that were interfaced to a Model 8610 GC from SRI Instruments. The SRI GC was equipped with a built-in Air Compressor and Hydrogen Generator that were used to supply gases for the GC column and detector flows. DET Report #41 presented data for both the situations of Hydrogen and Air as the GC carrier gas. Hydrogen allows the use of a wider range of column types without concern for destructive oxidation of the column coating, but its flow rate is very limited when an NPD is the detector. This report provides further discussion of the use of Hydrogen carrier with NPD and TID-1 detection on the "gasless" SRI GC.

Hydrogen flow rate is a critical parameter in the NPD. It is typically maintained in the range of 2 - 6 mL/min in order to prevent formation of a self sustaining flame when the NP Ion Source is heated sufficiently to ignite the H_2 - Air mixture. Gases on the SRI GC are pressure controlled, so the carrier flow depends on the length and diameter of the column, and the GC oven temperature. In the present work, we used a 6m x 0.53mm Rtx-TNT column (Restek), temperature programmed from 70 to 260°C at 18°C/min. For better control of the Hydrogen carrier, we inserted a restrictor (72 inch long x 0.62 inch O.D. x 0.005 inch I.D. stainless steel tubing coil from SRI) into the carrier gas line preceding the On-Column Injector. With this configuration, an H_2 carrier flow of 3 mL/min was achieved at 70°C with a supply of 12 psi from the H_2 generator. Detector Air flow was about 100 mL/min (supply pressure=3 psi).

Ion Source heating power is the other critical operating parameter in an NPD. Selective NP response turns on when the Ion Source is hot enough to ignite a boundary layer of H_2 - Air chemistry at the surface of the source. In this work, a stand-alone DET Current Supply was used to power the Ion Sources. For the NPD, the source heating current was 3.00 Amps, while for the TID-1 it was 2.50 Amps. Although the TID-1 ion source operated in the same H_2 - Air gas environment as the NP Ion Source (TID-4 type), the TID-1 source was not hot enough to ignite any gas phase chemistry. In this work, the existing SRI Electrometer was used to measure detector signals.

Figure 12 shows NPD and TID-1-Air chromatograms for a mixture of Explosive compounds (7 ng each component). The NPD detected all the explosives with an especially large signal for RDX (peak 6), while the TID-1 detector responded more selectively

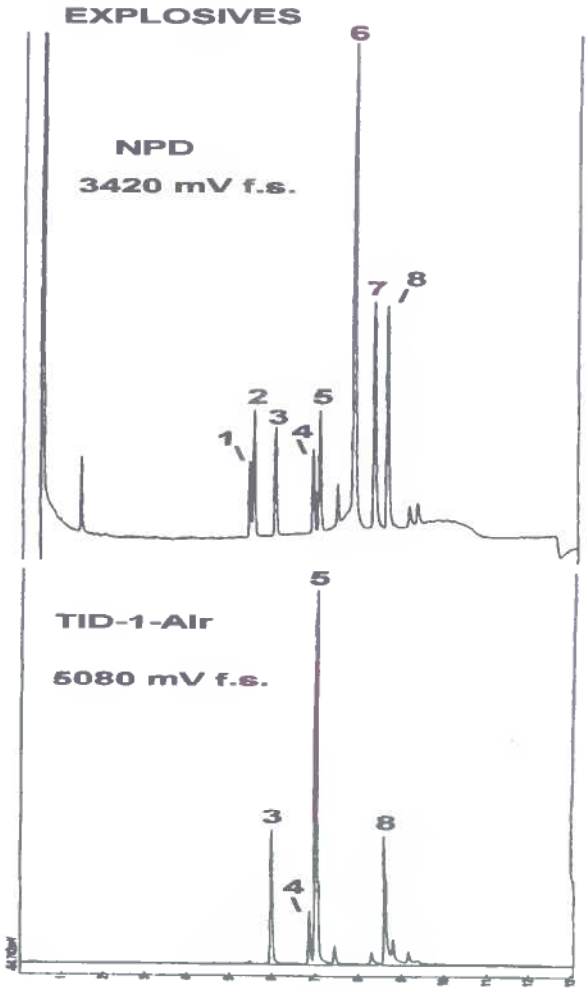

Figure 12. 7ng each, 1=1,3-Dinitrobenzene, 2=2,6-Dinitrotoluene, 3=2,4-Dinitrotoluene, 4=1,3,5-Trinitrobenzene, 5=2,4,6-Trinitrotoluene, 6=RDX, 7=4-Amino-2,6-dinitrotoluene, 8=2-Amino-4,6-dinitrotoluene. 6m Rtx-TNT.

with large responses to 2,4-Dinitrotoluene (peak 3), 1,3,5-Trinitrobenzene (peak 4), 2,4,6-Trinitrotoluene (peak 5), and 2-Amino-4,6-dinitrotoluene (peak 8).

Figure 13 shows NPD and TID-1-Air chromatograms of a sample mixture containing the two widely used Organophosphorus Pesticides, Methyl Parathion (MP) and Malahion (ML). The NPD detected both pesticides as well as the Nitrogen compound, Azobenzene (A). The sample also contained a large concentration of n-Heptadecane (C) to illustrate the selectivity of the detector. In contrast to the NPD, the TID-1 detector provided a much more selective response to just the Methyl Parathion.

In figure 13, the NPD exhibited more baseline drift with the column oven temperature program than did the TID-1 detector. This is a consequence of the greater sensitivity that the NPD has to changes in the magnitude of Hydrogen flow. We tried to minimize this NPD baseline change by accompanying the temperature program with pressure programming the Hydrogen carrier gas. However, the minimum programming rate of 0.1 psi/min allowed by the SRI data system overcompensated the Hydrogen flow. Furthermore, the NPD baseline change with temperature programming was not always the same. This also indicated an inadequacy of the SRI carrier gas controls to maintain the Hydrogen flow with the fine degree of precision required by an NPD. Baseline drift with oven temperature programming, therefore, appears to be a limiting factor affecting the ultimate detectivity achieved when using the NPD with a Hydrogen carrier gas.

The NPD data described in this report were generated with a TID-4 type Ion Source. This is a white colored ceramic coating. For Organophosphorus Pesticides such as in Figure 13, a better choice for the NPD Ion Source would be a TID-2 type which is a black colored ceramic. TID-2 is formulated to provide sharper Phosphorus peaks for longer operating times.

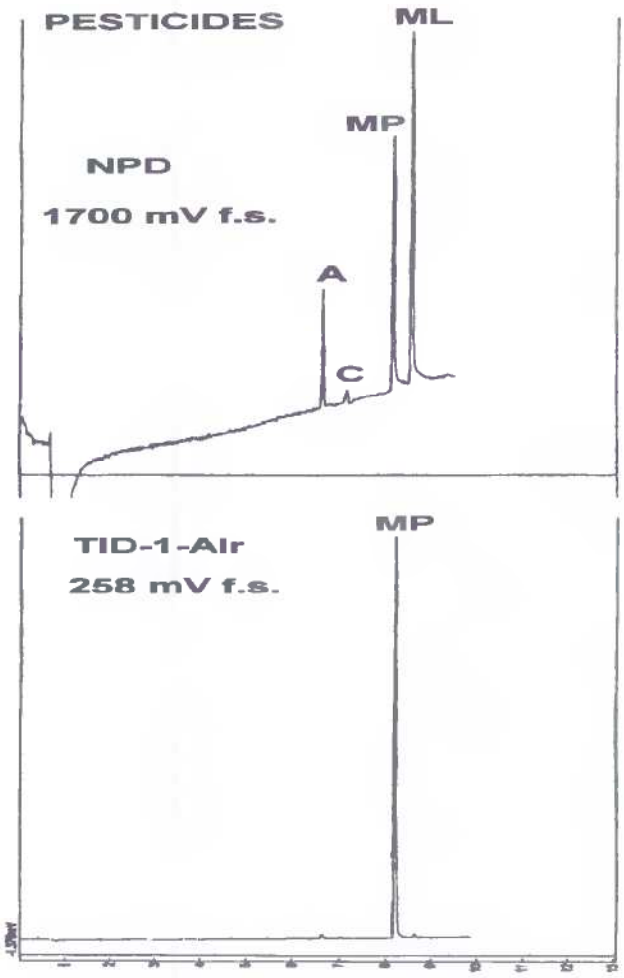

Figure 13. 1.2ng Azobenzene (A), 2400ng Heptadecane (C), 1.2ng Methyl Parathion (MP), and 2.4ng Malathion (ML). 6m x 0.53mm Rtx-TNT. Hydrogen carrier=12 psi (3mL/min at 70°C). Column temperature program 70 - 230°C at 18°C/min.

CHROMalytic TECHnology PTY LTD
ABN 14 643 445 058
Website: http://www.chromtech.net.au
PO Box 435, 232 Forest Rd, Boronia, Victoria 3155, Australia
Tel: +61 3 9762 2034 Fax: +61 3 9761 1169 E-mail: sales@chromtech.net.au

DET REPORT

NO. 42 AUGUST 2001

Figure 14 shows NPD and TID-1-Air chromatograms for a sample mixture containing Methamphetamine (1), Nicotine (2), Cotinine (3), Cocaine (4), and Diazepam (5). These drug compounds all contained N-atoms, so the NPD responded with a large response to all 5 components. The TID-1 detector had a much lower selective response to Cotinine, Cocaine, and Diazepam, plus a few other peaks not detected by the NPD. With respect to the selective response of TID-1-Air, it may be relevant that the three compounds detected all contained O-atoms as well N-atoms, whereas the Methamphetamine and Nicotine components did not.

The same 6m x 0.53mm Rtx-TNT column was used in the SRI GC for all the data in this report. This short column exacerbated the problem of controlling the Hydrogen carrier flow with sufficient precision for the NPD. A longer column could provide enough additional flow restriction to allow pressure programming to be used effectively to reduce NPD baseline drift during a temperature program. In any case, an additional flow restrictor tubing inserted in the carrier gas such as described earlier, helps in maintaining NPD-Hydrogen carrier pressures within the most controllable range of the SRI electronics. To ensure that the H_2 carrier flow is in the correct range for NPD operation, we believe it is necessary to measure the flow at the detector jet structure whenever the column is changed to a different length or diameter. In this work, we established that the H_2 carrier flow was in the NPD range of 2 - 6 mL/min with the GC column at its lowest operating temperature. This ensured that the H_2 flow would decrease and stay within the NPD range as the column temperature increased with the column head pressure held constant.

TID-1 detection with a Hydrogen carrier gas is much less restrictive than NPD detection because the TID-1 source is not hot enough to ignite the H_2 - Air chemistry. Also, NPD operation with an Air carrier rather than Hydrogen is less complex because the H_2 then can be supplied as a fixed pressure detector gas that does not change flow as the column oven is temperature programmed. DET Report #41 illustrated such an NPD application. In the present work, the detector gas line that normally is used for H_2 was capped.

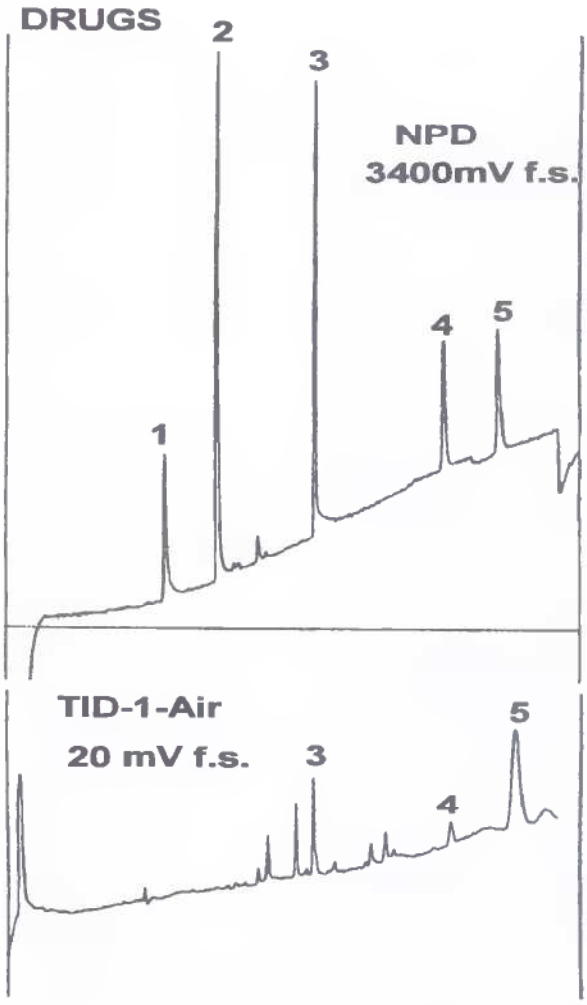

Figure 12. 6ng each, 1=Methamphetamine, 2=Nicotine, 3=Cotinine, 4=Cocaine; 3ng of 5=Diazepam in a Methanol solvent. 0.6µL injected On-Column.
6m x 0.53mm x 1.5µm Rtx-TNT. 70-260°C at 18°C/min, 260°C-3min. DET NPD and TID-1 on SRI GC with Hydrogen carrier (12psi with added 72in. X .005in. X .062in. carrier restrictor). Detector Air=3psi (100mL/min). Detector = 290°C.