THEORY AND OPERATION

OF THE

NPD/TID/CFID DETECTORS

FTID DETECTOR

REMOTE FID DETECTOR

TANDEM TID DETECTOR

FID DETECTOR

NPD	-	NITROGEN PHOSPHORUS DETECTOR
TID	-	THERMIONIC IONIZATION DETECTOR
CFID	-	CATALYTIC FLAME IONIZATION DETECTOR
FTID	-	FLAME THERMIONIC IONIZATION DETECTOR

DET
innovations in chemical detection

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I. GENERAL THEORY

A. BASIC DETECTOR COMPONENTS

The basic configuration of DET detectors is a cylindrically-shaped thermionic/catalytic source positioned on the axis of a cylindrical collector electrode. This configuration provides a stream-lined gas flow through the detection volume, and a radial-shaped electrical field for efficient ion collection. The thermionic source is heated by a constant current power supply, and is biased at a negative voltage with respect to the collector. Negative ionization current is measured with a conventional electrometer. The detector generally mounts onto an NPD or FID type detector base so that at least two sources of detector gases can be supplied. One of the detector gases, and often a third "Makeup" gas, are introduced through the center of a sample conduit tubing, along with the sample carrier gas from the GC. The remaining detector gas sweeps the outer diameter of the sample conduit tubing. In many cases, a conventional FID flame tip suffices as the sample conduit.

The most important parameters in this detector are the composition of the surface of the source, the temperature of the source, the composition of gases surrounding the source, and the magnitude of polarization between the source and collector. Entirely different types of detector responses are obtained through variations in any or all of these key parameters. Consequently, the basic detector hardware provides the capability for a whole family of operating modes.

B. THERMIONIC IONIZATION DETECTION (TID)

Samples impact the heated, alkali activated ceramic surface of the source, and are ionized by the extraction of electrical charge from the surface. This surface ionization process is controlled by the surface work function, the surface temperature, and the composition of gases surrounding the surface. N,P specificity is obtained from a very hot surface of moderate work function operating in a chemically active environment consisting of a dilute concentration of H_2 mixed with air (TID-2-H_2/Air). Very high specificity and sensitivity to certain compounds containing electronegative functional groups is obtained from a low work function source operated at moderate temperatures in an inert environment of pure N_2 (TID-1-N_2), or in an oxygen containing environment (TID-1-Air).

C. CATALYTIC FLAME IONIZATION DETECTION (CFID)

A source comprised of ceramic and a non-alkali additive serves as a combination ignitor, polarizer, and catalytic surface in a H_2/air flame environment. In this CFID, the ionization occurs primarily in a gas phase process, and universal responses similar to those of an FID are obtained. The catalytic surface aids in the combustion process, and provides enhanced responses to certain compounds normally having reduced responses in conventional FIDs.

D. FLAME THERMIONIC IONIZATION DETECTION (FTID)

An alkali activated ceramic source of low work function is operated at moderate temperatures in the effluent stream of a H_2/Air flame. Unlike the CFID, ionization in the flame environment is normally not measured. Instead, the neutral products of combustion are selectively re-ionized by the thermionic transducer placed downstream of the flame. The FTID provides specific responses to heteroatom compounds which yield electronegative products of combustion, especially nitrogen or halogen compounds.

E. REMOTE FLAME IONIZATION DETECTION (REMOTE FID)

Similar to an FTID, a ceramic source structure and ionization collector are located several centimeters downstream of a flame. Unlike an FTID, the thermionic source is operated in a manner that does **not** produce any additional ionization beyond that originally produced in the flame. Instead, the source functions as a polarizer to produce collection of long lived negative ions emanating from the flame. The REMOTE FID provides specific responses only to compounds which produce very stable negative ions in combustion. Examples are the specific detection of lead, tin, or phosphorus compounds.

F. TANDEM TID

TANDEM TID refers to a series combination of two independently controlled thermionic ionization detection stages. Each stage provides a different response, and both responses are obtained simultaneously for each sample compound.

G. FLAME IONIZATION DETECTION (FID)

FID detection of all organic compounds is achieved by using an uncoated loop of Pt/Rh wire in place of the thermionic source.

H. UNIQUE LAYERED CONSTRUCTION OF THE SOURCE

In the thermionic ionization process, charge lost from the surface of the thermionic source (I_2) must be replaced by a migration of charge through the body of the source (I_1). The present TID sources have a separate non-alkali/ceramic sublayer through which charge migration occurs, and an alkali/ceramic surface layer optimized to provide the type of selective surface ionization desired. There are currently available 7 different type thermionic sources identified as TID-1, TID-2, TID-3, TID-4, TID-5, TID-6, and CFID; and an FID ignitor/polarizer probe which is an uncoated Pt/Rh wire.

I. GENERAL OPERATING CHARACTERISTICS

The TID, CFID, FTID and REMOTE FID are mass flow rate sensitive detectors rather than concentration sensitive detectors. This means that relatively high flows of detector gases can be used to sweep the detector volume without causing reduced response due to sample dilution effects. The composition and flow rates of detector gases, however, do influence the surface temperature of the source. In most modes of operation, the source temperature will be in the general range of 400 - 800°C, whereas the surrounding detector tower will be at a wall temperature of 100 - 400°C as set by the detector heater block controls on the GC. The resultant source temperature is a balance of the electrical heat input to the source and heat losses due to conduction and convection through the gases flowing past the source. Therefore, the source temperature is dependent on the magnitude of source heating current, the detector heater block temperature, the thermal conductivity of the gas mixture flowing past the source, and the magnitude of the total gas flow through the detector. In the CFID, FTID, and REMOTE FID, the source is also heated to some extent by the H_2/Air flame burning around and/or beneath the source.

From the above considerations, the following general operating characteristics can be expected:

1.) At any fixed set of gas flows and detector heater block temperature, the principal means of varying source temperature is via the magnitude of source heating current.

2.) If the gas flows or detector heater block temperature are changed, a readjustment of source heating current restores the source to the same surface temperature it had before the change. Often the magnitude of detector background signal or the response to a standard sample can serve as a guide to the correct readjustment of source heating current.

3.) Helium has a much higher thermal conductivity than nitrogen, so the use of helium as the GC carrier gas generally requires higher settings of source heating current than when nitrogen is used.

4.) As the total gas flow through the detector is increased, generally expect to supply more heating current to obtain the same source temperature. The present detector has a small internal volume, so for most TID modes a total gas flow of 80 - 120mL/min is adequate. For the CFID, FTID, and REMOTE FID, typical gas flows are 200 - 250mL/min.

5.) As the detector heater block temperature is increased, expect to supply less heating current to the source to obtain the same source temperature. The detector has been designed to operate for extended periods of time at detector heater block temperatures of 400°C. As a general rule, it is best to operate the detector heater block temperature as high as is allowable by the application. This minimizes the temperature gradient between the source and the surrounding wall, and helps minimize detector contamination.