

# Review of Photoionization Detection in Gas Chromatography: The First Decade

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ment of ternary electrochemical sensors. Mr. Driscoll also developed a novel vacuum UV photodiode which led to the development of a far UV absorbance detector for gas chromatography.

## Introduction

Photoionization, as a means of detection, has been with us for about 25 years. Robinson (1) first reported on the development of a photoionization detector in 1957. At the same time, groups in various parts of the world (2) were working on the development of flame ionization techniques. This latter technique became very popular and was rather quickly licensed to a number of commercial gas chromatography (GC) manufacturers since the detector was very sensitive and easy to build. Lovelock (3) became interested in the photoionization technique and published a review of ionization techniques in 1961 (4). This included the flame ionization detector (FID), the photoionization detector (PID), cross section, and electron capture detection (ECD). The FID has become the most popular in the group as a result of its wide dynamic range, its selective response to organic compounds, sensitivity, and its simplicity of construction. While the ECD has also maintained a significant place as a selective detector for halogenated species, the cross section detector has been forgotten.

During the 1960s and 1970s, a variety of papers (5-9) on photoionization were published using glow or microwave discharges as the ionizing source. Both types involved flowing high purity helium or argon through an electrical or microwave discharge to generate the source of energetic photons required to

ionize the sample. With these early PIDs, the light source and ion chamber were not separated and, as a result, neither section could be optimized. The maximum lamp intensity occurs at low pressures while the maximum sensitivity for the ion chamber is at or near atmospheric. As a result, these early detectors were very pressure (or flow rate) dependent. In general, these detectors were difficult to operate, mechanically complex, unstable, and required a vacuum pump. The glow discharge detector had the continual problem of column bleed collecting on the discharge electrodes, resulting in problems with igniting the lamp. It is no wonder that the PID was replaced with the FID during the 1960s. By the late 1960s most researchers agreed that photoionization was not a viable analytical technique. The FID, in the meantime, had become firmly entrenched as the detector of choice for analysis of carbon compounds.

In the period from 1973 to 1974, a major breakthrough in photoionization technology (separating the ion chamber from the UV lamp) was reported by Driscoll and Spaziani (10), Sevcik and Krysl (11), as well as Ostojic and Sternberg (12). This design also eliminated many of the deficiencies of the glow discharge detector by allowing the ionization chamber to be operated at atmospheric pressure and the lamp to be maintained at low pressures. This improved the sensitivity and simplified the operation of the detector. Now, after half a decade, photoionization appeared to be on the upswing again.

The PID described by Driscoll and Spaziani (13) was offered commercially by HNU Systems in the spring of 1976. Since this was the first commercial PID, it attracted a great deal of interest because of its reported 50-fold improvement in sensitivity over the FID for aromatic hydrocarbons. Other features of the new PID that were interesting were its response to inorganic compounds, and its non-destructive nature. Within two years, however, the PID was replaced with a new model (14) that eliminated some of the deficiencies, such as temperature limitations and decomposition of thermally labile compounds on the inlet, which were inherent in the first PID.

In the following sections, some recent applications of the PID for analysis of organic and inorganic compounds will be briefly described. All applications and additional references in the text will be to the PIDs with the separated lamp and ionization chambers. The period covered will be from 1976 to mid-1985 or, the first decade of literature after the commercialization of the PID; hence, the title for this paper.

## Literature Sources

A computer search was conducted through BRS/After Dark utilizing a Model 200 portable computer (Tandy) with a built-in modem. The first database searched was the American Chemical Society Primary Journal Database, which provides abstracts and complete texts for 18 primary journals, covering the period from 1980 to June 1985. The keywords used included photoionization and gas chromatography, or photoionization detectors and gas chromatography. Following the first search, the Chemical Abstracts Database was searched for the time period from 1980 to June 1985. No abstracts were available on-line with this database. In addition, the pre-1980 references were obtained by a manual search of chemical abstracts, and personal files were searched to provide some unpublished works.

## Organic Analysis

The first applications of the PID were related to the improvement in sensitivity compared to the FID. Tyre (15) took advantage of the PID's concentration-sensitive response (e.g.,  $C = 1/F$ , where  $F$  = carrier flow). By reducing the carrier flow to about 1 cc/min, Tyre was able to detect ppt levels of vinyl chloride in the atmosphere. For this application, the PID was 1,000 times more sensitive than the FID. Hester and Meyer (16) used a PID and a technique similar to Tyre's to detect sub-ppb levels of benzene and toluene in the atmosphere. They reported that aromatic hydrocarbons could be detected at concentrations of 0.3 ppb with a 2:1 signal-to-noise level.

Smith and Krause (17) used a PID for analysis of carbon disulfide desorbed from charcoal tubes. The PID was found to be easier to use, more sensitive, and have a wider dynamic range than the flame photometric detector (FPD). Although the PID was not sulfur specific, these features clearly compensated for that lack. Kramer and Wechter (18) were involved in the analysis of gas cylinders containing reduced sulfur compounds at levels from low ppb to high ppm. They used the FPD for the low levels but it saturated at 1 to 5 ppm and a second technique (near UV absorbance) was required for the high levels. The PID was found to be useful over the entire range of approximately 6 to 7 decades for  $H_2S$ ,  $CS_2$ , and mercaptans. Zarembo (19) was interested in the PID for its lack of specificity and because of its response to sulfur, nitrogen, and carbon compounds. One detector could now be used to replace three selective detectors.

Some typical applications were described for a new PID which included detection of both organic and inorganic gases. Typical detection limits were in the low picogram range for organics and certain inorganic compounds, such as hydrogen sulfide, iodine, and phosphine. Driscoll (20,21) described a PID which was 30 times more sensitive than an FID, had a detection limit of 2 pg for benzene and was a carbon counter like the FID. Oyler et al. (22) described a method using high performance liquid chromatography (HPLC) for an initial separation of complex hydrocarbon mixtures, followed by injection of the HPLC fractions into a gas chromatograph to further separate the polyaromatic hydrocarbons (PAH). The detection limits for these PAHs were found to be in the 50- to 100-pg range or about 10 to 40 times lower than for an FID. In addition, the PID showed negligible response to acetonitrile and water, the mobile phase in the HPLC separation.

In 1978, Driscoll et al. (14) described a new high temperature PID. The earlier detector (13) had been limited to temperatures of 200°C because of the Teflon® ion chamber. The new PID had a lower dead volume (200  $\mu$ l), higher operating temperature (300°C), and was more inert. Jennings et al. (23,24) analyzed flavor essences by capillary GC and utilized the non-destructive aspect of the PID to sniff flavor essences at the detector exit. With an FID, a splitter is required since the detector is destructive. They also described a method to switch the PID effluent into a second capillary column of different polarity and higher resolution, to confirm the identity of various flavor essences. Levins et al. (25) developed a method for analysis of tetraethyllead (TEL) in work atmospheres. The TEL was collected on an ion exchange resin, desorbed with pentane, and analyzed by GC with a PID.

One of the more important applications for the PID has been the analysis of organics in drinking water. A method (EPA 602) for analysis of purgeable aromatics in drinking water was described in the Federal Register (26). The procedure involves concentrating the water sample via the purge-and-trap technique. The sample is then injected into a GC and analyzed by PID. The Federal Register specifies the HNU PID. The PID was chosen for this application because of its increased specificity over the FID. A method for analysis of drugs described by Jaramillo and Driscoll (27) was reported to be 8 to 16 times more sensitive for barbiturates than the FID. The PID correlated well ( $r^2 = 0.99$ ) with the FID on extracted serum samples and produced less background than the FID for most samples. Some of the advantages for drug analysis include simplicity, lack of solvent response, stability, and the elimination of the hydrogen fuel. Jaramillo and Driscoll (28) described some modifications to the PID that improved its performance on capillary columns. The major modifications involved changing the seal material, addition of 10 to 15 ml/min of make up gas, and the insertion of the capillary column up to the end of the inlet tube. Once these modifications were made, the performance nearly matched that of the FID. Langhorst and Nestruck (29) described a method for collection and analysis of mono- to hexachlorobenzene isomers in air or urine. The air samples were collected on XAD-2 resin and desorbed with carbon tetrachloride. The recoveries for the chlorobenzenes were  $83 \pm 12\%$  for concentrations between 1 and 500 ppb (ng/g). Meili et al. (30) utilized capillary GC to analyze nitrosamines in meats. They found the PID response to dimethylnitrosamine and ethyl-butyl-nitrosamine to be linear with detection limits of 50 to 100 pg. A Sep-Pak cleanup helped significantly but a more extensive procedure was required for accurate quantitation of the nitrosamines. Nestruck et al. (31) described some chemically bonded supports which can be used to improve the performance of the PID and other detectors. Freedman and Worker (32) developed a method for analysis of oil in breathing air by PID. They were able to detect 2 ppb of oil vapor in a  $CO_2$  stream on a continuous basis. Narang and Bush (33) developed a stripping method for removing chloroalkenes, vinyl chloride, and arenes from water. They were then adsorbed on Porapak N, eluted with methanol and analyzed by GC/PID. They obtained detection limits of 1  $\mu$ g/l for all the compounds. They demonstrated that the PID provided good response to species with low electron absorbing properties, reliable results, and detection limits well below the conventional FID. Freedman (34) evaluated the performance of the PID with respect to developing a theoretical model for predicting response. The PID response was shown to be dependent upon molar response and ionization potential. He also stated

that there is no relationship between PID response and carbon number. However, Langhorst (35), in a more detailed study, found that the sensitivity does increase with carbon number. Other findings were that for substituted benzenes, ring activators increase sensitivity and ring deactivators decrease sensitivity with the exception of halogenated benzenes. Langhorst (35) determined the response factors for several hundred compounds. This work provides the most extensive treatment for the 10.2 eV PID. Driscoll et al. (36) described a GC with an integrated PID that enables detectors (PID/FID or PID/ECD) to be run in series. This technique for sample identification will be discussed in detail in the next section. Driscoll (37) and Zepeck (38) describe some applications of the PID in industrial hygiene including analysis of ppb levels of vinyl chloride and inorganic gases such as  $H_2S$  and  $PH_3$ . Low MW hydrocarbons such as  $CCl_4$  were detected with an 11.7 eV lamp in the PID since these species have higher ionization potentials and could not be detected with the 10.2 eV lamp. Ho et al. (39,40) developed a coated piezoelectric crystal detector for toluene and verified the response by comparison with the PID. Hembree (41) discussed the use of a non-destructive detector such as the PID to eliminate the splitter/FID combination in between the Fourier transform infrared (FTIR) capillary GC system. Stein and Narang (42) used a PID to improve the sensitivity of vinyl chloride in water. The PID was found to be more sensitive than the Hall, the FID, or the ECD for analysis of aqueous headspace samples. Senum (43) found the PID response to be dependent upon the carrier gas. The optimum response was produced with argon. Klimak and Wessel (44) constructed a PID with a laser light source. They used a tunable source to differentiate between anthracene and phenanthrene, which were not resolved on the GC column. They also indicated that two-photon photoionization was more efficient than single-photon photoionization, although their data did not prove this to be the case. If the cost and complexity of laser systems can be significantly reduced, this technique should prove very useful in the future. Van Tassel et al. (45) described a method for collection and analysis of arenes and volatile haloorganic compounds in air. The samples were collected on Porapak N, desorbed with methanol, and analyzed with an ECD or PID. A 20-liter air sample yielded detection limits of 1 and  $5 \mu g/m^3$  for the ECD and PID, respectively. Hewitt and Driscoll (46) and Driscoll et al. (47) described automatic GCs which detect ppb levels of organic compounds on a continuous basis via PID. Stein and Narang (48) describe a method for determination of mercaptans at  $\mu g/m^3$  levels in air by PID. With a 5-ml sample loop, the PID detection limit of 2 to  $4 \mu g/m^3$  was found to be the same as the odor threshold. Driscoll (49) developed a method for identification of hydrocarbons using capillary GC and three lamps (9.5, 10.2, and 11.7 eV) for the PID. Alkanes, alkenes, aromatics, and polyaromatics could be identified at nanogram levels by determining the response ratios of the sample with the various lamps. Yancey (50) evaluated infrared and photoionization detectors for analysis of new synthetic crude oils. The PID was found to be capable of determining aromatic hydrocarbons in complex aliphatic species. Rigin et al. (51) developed methodology for analysis of anilines in waste water. During this process, they evaluated the nitrogen phosphorus detector (NPD), the Hall detector, and the PID with 10.2 and 9.5 eV lamps. The most sensitive detector was the PID (10.2) but it did not have sufficient selectivity. The PID (9.5) had more selectivity, but the sensitivity to nitro anilines was not adequate. The NPD was selected for the analysis. Tanaka and Shinozaka (52) used GC/PID to analyze aromatic hydrocar-

bons in the atmosphere. Lewis, Mulik, Coutant et al. (53,54) used a PID for analysis of hydrocarbons thermally desorbed from passive sampling devices. Becker et al. (55) describe calibration techniques which can be used to calibrate PID-based instruments. Driscoll and Krull (56) describe chemically bonded supports which can be utilized to improve the performance of the PID and other chromatographic detectors. Levine et al. (57) used a PID to determine the cross contamination of water samples taken for analysis of purgeable organics. Collins and Barker (58) describe a portable PID-based GC for monitoring of ethylene oxide and ethylene dibromide in air. Clark et al. (59) compared direct GC/PID to samples collected on Tenax tubes. The minimum detectable quantity (MDQ) for the PID was 0.3 ppb for benzene and toluene but 1 ppb for xylene. Good agreement was obtained between the two methods for the above compounds, but inconsistencies were found for the higher alkyl benzenes. Kirschen and Wood (60) used a PID and Hall detector to measure aromatic hydrocarbons and chloroalkanes in water samples. A purge-and-trap technique was utilized to remove the hydrocarbons from water. Baim et al. (61,62) reported on a photoionization source for determining ion mobilities. Kapilla et al. (63) described a PID and ECD combined. A readily photoionizable gas was bled into the detector when operated in the ECD mode to provide a standing current. With the UV source, a linear response was obtained from the pg to  $\mu g$  range. One difficulty with this technique is that strongly photoionizable species will give large negative peaks. Dumas and Bond (64) report on the detection of sub-ppb levels of methyl bromide with a PID. Bellar and Lichtenberg (65) report on a method (EPA 503) for determination of volatile aromatic compounds in drinking water and raw source water. These compounds were extracted via the purge-and-trap technique, then analyzed by GC/PID. The optimum conditions are given for over 30 common organics. Detection limits ranged from 0.002 to  $0.02 \mu g/l$ . Significant losses were noted on storage of nonpreserved water samples. Recoveries are reported. Amin and Narang (66) report on a method for the determination of volatile organics in sediment at the ng/g level by GC/PID. The volatile organics were stripped from the sediment, concentrated on Porapak N and eluted with methanol. The detection limit for PID-active compounds was 7 ng/g and 1 ng/g for ECD-active compounds. Dumas (67) compared PID, FID, and thermal conductivity detectors (TCD) for the determination of formaldehyde. Davenport and Adlard (68) reported on improvements made to a commercial PID to improve the performance. They found that the PID had a high sensitivity and selectivity for classes of compounds such as aromatics and olefins. A description of the photoionization process is also given. Driscoll et al. (69-71) describe continuous monitoring instruments for ethylene oxide which comply with the new federal Occupational Safety and Health Administration (OSHA) standards and use GC/PID for detection. Applications for PID (10.2 and 11.7 eV) are compared to the FID for sulfur compounds and to the ECD for pesticides.

### Detectors in Series

A technique was reported (72) seven years ago which utilized the unique response ratio of two detectors, FID and PID, to identify hydrocarbon structures. The PID (10.2) response was found to be a function of the electronic structure (pi electrons were ionized more efficiently than sigma electrons of the hydro-

carbon; as a consequence, it provided discriminative response). The PID response was compared to the FID which has a homogeneous response for hydrocarbons (the response depends mainly on the number of carbon atoms, not on bonding environment, e.g., sigma or pi electrons). This technique was first used by Driscoll et al. (72) to identify aromatic, olefinic, and aliphatic hydrocarbons in a synthetic natural gas feedstock. Kapilla and Vogt (73) used the PID/FID technique to classify eluted peaks from coal gasification samples into aromatic or aliphatic hydrocarbons. Driscoll (49) reported on the use of a PID with three lamps (9.5, 10.2, and 11.7 eV) to identify hydrocarbons. In this instance, the 11.7 PID replaced the FID, and the 9.5 PID provided additional selectivity and the ability to identify some polyaromatic hydrocarbons. Of course, it is possible to run two or more PIDs in series. Driscoll and Hewitt (74) have reported on the use of the PID/FID technique for identification of hazardous waste components. Nutmagal et al. (75) reported on the use of PID/FID to classify hydrocarbons in ambient air. The method was used to determine the aromatic, olefinic, and aliphatic weight fractions in ambient air. Bonelli and Kebbekus (76) reported on a three detector system for identification of specific organic compounds based on the response ratios of the ECD, PID, and FID. The PID/FID system was used with capillary GC to identify C<sub>2</sub>-C<sub>10</sub> hydrocarbons. Cox and Earp (77) used subambient programming and measured the retention times and normalized detector response ratios for 143 compounds, including aromatics, olefins, and alkanes. Towns and Driscoll (78) detected and identified amines using PID/NPD. The use of PID (10.2)/NPD in a qualitative sense will provide all peaks with nitrogen. For structural purposes, di- or triamines, aliphatic, or aromatic amines can be determined by their ratios. The PID (8.3)/NPD response ratios can be utilized to differentiate between primary, secondary, and tertiary amines. Krull et al. (79) combined PID and ECD detectors as a means for differentiating between PAHs from nitro PAHs. A similar technique was used by Krull et al. (80) for the identification of organic nitro compounds and explosives. Driscoll et al. (81,82) combined the PID with the far UV absorbance detector for identification and analysis of sulfur compounds, formalin, degreasing solvents, etc. Krull et al. (83) reported on the use of multiple detectors (including the PID) in gas chromatography. They described in-series and parallel detection, data acquisition, and final manipulation.

### Derivatization

In addition to the direct determinations with the PID, there also exists the possibility to convert a species which has a low or lack of response on the PID to another species which will respond. For example, Lindqvist (84) converted nitric acid to nitric oxide and detected it by PID.

Using this analytical technique, he developed a method for analysis of nitric acid aerosol in the atmosphere. Lindqvist (85) was also able to determine sulfuric acid aerosol in the atmosphere by conversion to hydrogen sulfide and analysis with the PID. Krull et al. (86) derivatized alcohols with flophemysyl chloride and compared the PID results with ECD and underivatized alcohols. The MDQs for the derivatized alcohols varied from 20 to 47 pg for the PID and 14 to 58 pg for the ECD. The improvement in MDQs for the PID was between two and three orders of magnitude. A similar approach was

reported for drugs and steroids (87). MDQs in the low pg range were obtained for both the drugs and the steroids.

### Past and Future Prospects

The past decade has seen a tremendous increase in the use of the PID as a routine detector in GC. Initially, the PID was used because of its increased sensitivity compared to the FID. After a few years, the selectivity of the PID became important, e.g., EPA Method 602, and the use of PID in series with another detector became more common. Some of the unique characteristics of the PID have been utilized to provide an extremely useful and reliable detector for GC. In the next few years, or perhaps decade, the PID should become one of the most widely used selective detectors in GC as a result of its versatility and performance.

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