

# Platinum in Explosives Detectors

## UNIQUE SURFACE PROPERTIES SUCCESSFULLY UTILISED

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Over the last decade there has been a drastic increase in the use of explosives by terrorist groups of various political complexions, both in this country and abroad. This has led to a demand for instruments capable of detecting the presence of concealed explosives and also the traces left behind by explosives in areas which have been used for their storage, handling or manufacture.

With trivial exceptions, the main line of successful attack on this problem has been the detection of the vapour emanating from solid explosive. Most explosives have a low but finite vapour pressure and the presence of concealed material will be indicated by a low concentration of vapour in its proximity.

The concentrations involved are extremely dependent on the physical circumstances of concealment, but are always low compared with the levels usually encountered in gas analysis work. Experience has shown that to be successful in a field situation an explosives detector must be able to respond to concentrations by weight of explosive vapour in air of the order of one part per billion (1 in  $10^9$ ). Furthermore, the detector must not respond to any of the many natural and man-made vapours which are present in the environment at this level and above.

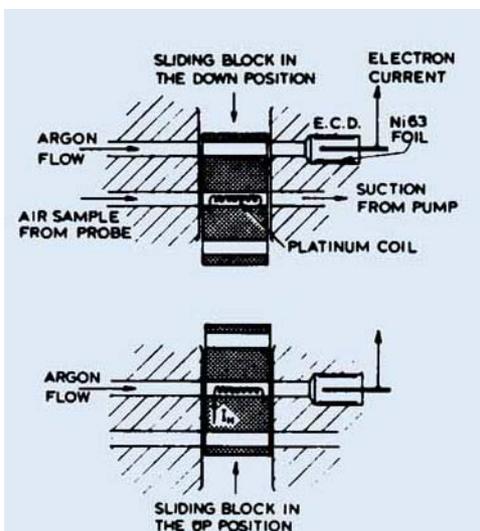
### Method of Detection

Most detectors so far developed have made use of a detection device derived from gas-liquid chromatography practice and known as the electron capture detector (usually abbreviated to E.C.D.). In this device a weak radioactive source, typically Ni63, bombards a volume of gas—which may be argon,

nitrogen or helium—with low energy  $\beta$  particles. The gas molecules are ionised and a concentration of free electrons is built up. The electrons are periodically stripped out of the ionised gas by the application of a short positive pulse to a suitable electrode; the average current drawn from this electrode is then a measure of the concentration of electrons existing at the moment of application of the pulse.

If molecules of another material are now fed into the cell, some of the free electrons may be captured by these molecules to form negative ions. Whether or not this happens for a particular substance depends on an inherent property of that substance, known as its electron affinity. This property varies over a wide range; some substances, for example  $SF_6$ , are extremely active as electron capturers, while others, such as oxygen, are much less so.

If some electrons have been captured, the free electron concentration in the cell will fall, and the periodic stripping will result in a lower mean current. In practice, extremely small concentrations of an electron-capturing species will give appreciable falls in the collected current. Why this should be so may not be immediately clear, but a consideration of the numbers involved will help understanding. The numerical concentration of the free electrons relative to the gas molecules is very low—perhaps only 1 in  $10^8$ . If the numerical concentration of the introduced electron capturing species is, say, 1 in  $10^{10}$  this should be related not to the total number of molecules present but to the far smaller free electron population—a ratio of one hundred to one.



**Fig. 1** Two stages in the sampling operation. First, with the block in the down position, air is drawn from the sampling point and passes over the platinum coil where vapour from the air sample is adsorbed. Then, with the block in the up position, a flow of argon passes over the coil and a current  $I_H$  is applied; this causes any adsorbed material to be desorbed and pass with the argon into the electron capture device where the analysis takes place

Fortunately, most explosive materials are strongly electron-capturing and respond well in an E.C.D. The basic problem, however, is that they are present as vapours in an oxygen rich environment, and oxygen, though weakly electron-capturing, will in high concentration saturate the response of the E.C.D. The vapour molecules must somehow be removed from the air and transferred to a passive carrier gas.

Historically the first way of achieving this was to use an organic membrane selectively permeable to organic molecules and relatively impermeable to oxygen. However, it is difficult to produce membrane separation systems with a high degree of specificity to explosives vapours, with the result that

equipments based on this principle can give rise to false responses from such interfering vapours as perchloroethylene—a common dry-cleaning solvent—and certain perfumes, toiletries and medicants. By using the unique surface properties of platinum, Higgs and his co-workers in the Procurement Executive of the Ministry of Defence were largely able to avoid this problem.

### Selective Vapour Adsorber

Referring to Figure 1, Higgs' system, shown here in idealised form, uses a coil of platinum wire as a selective adsorber of vapours from an air sample. In the sampling phase, air from the sampling point is drawn via a probe over the platinum coil for two seconds. The coil is then transferred from the air stream into a flow of argon which passes through an E.C.D. A heating current is then passed through the coil and any adsorbed material is desorbed into the stream of argon. Any electron capturing species in the desorbed material will cause a drop in the current drawn from the E.C.D. The presence of appreciable amounts of explosive vapour in the desorbed sample causes an enhanced drop in the E.C.D. current; this is monitored by electronic circuitry and used to sound an alarm.

The advantages of platinum in this application are threefold. The first is the high capacity of the surface for adsorbed material. The second, and most remarkable, is the extremely high specificity of the surface for the vapours of interest against common contaminating substances which might cause false alarms. While the as-drawn surface of the wire shows this property to some degree, surface treatment procedures developed by Higgs enhance the effect to an extraordinary degree. The third advantage is the resistance of the treated platinum surface to degradation due to the many and varied vapours which are drawn over it in use. The working life of the surface may be several hundred hours.

A practical detection instrument must be

*Fig. 2 The explosives detector, which only weighs 10 kg, is housed in a brief case. The probe enables air from the area of interest to be sampled and passed for analysis into the electron capture detector where the presence of any explosive vapour is detected electronically. The complete sampling and analysing cycle takes only three and a half seconds*



sensitive, but must also operate satisfactorily under gross overload both of explosive vapour, which it must detect, and of contaminant, which it must not. Under such conditions an excess of material is transferred into the carrier gas stream by mechanisms other than surface adsorption on the platinum. This can result in partial or complete loss of electron current in the E.C.D., due to overload with electron capturing species. To overcome this problem the electronics have been designed to recognise the condition and delay heating of the platinum collector until the E.C.D.

current has recovered almost to its normal value. Heating of the platinum will then produce a very large drop in E.C.D. current if explosive material is present; if only contaminant is present, almost no drop occurs. By this artifice the equipment is able to distinguish between explosive vapour and contaminant vapour which may be several orders of magnitude higher in concentration.

The first engineered detector using this principle was developed by Pye Dynamics Limited with the support of the Procurement

Executive, of the Ministry of Defence. It weighed 18 kg and took one sample every sixteen seconds in a cycle of operations which involved the use of motor driven valves.

### Cycle Time Reduced

Although later developments reduced the cycle time of this equipment to eight seconds this was still considered too slow, and Pye Dynamics undertook the development of an improved version for civil use. Gas switching in this new instrument is by fluid logic and all the active components, including the E.C.D., are incorporated in drillings in a block of P.T.F.E. Attention to detail design

has brought the weight down to 10 kg, and the equipment is now housed in an easily carried unobtrusive brief case (Figure 2). Improved electronics enables the response to be shown on a digital display. Most important of all, however, is the cycle time; this is now three and a half seconds, made up of two seconds for sampling and only one and a half seconds for analysis.

It is a sorry commentary on the state of the world today that this equipment is meeting with substantial commercial success. It is also true that its excellent performance depends entirely on the unique surface properties of a short length of platinum wire.

## Control of the Penetration of Platinum into Alumina Pellets

Small cylindrical pellets of alumina are frequently used as supports for noble metals to be used as catalysts in fixed bed reactors. It has been known for some years that the observed efficiency of such catalysts depends upon the location of the metal within the pellet, and for many reactions it has been believed that a very thin layer of metal close to the pellet's surface produces optimum results. Recent theoretical discussions (1, 2) have led to a clearer understanding of the importance of the concentration profile of the metal through the pellet: if the reaction has an overall positive order it is better to have the metal near the surface, but if it has an overall negative order then it is preferable to concentrate the metal towards the centre of the pellet.

Such theoretical considerations are of little value unless they can be put to experimental test, and for this purpose a means of controlling the location of the metal within the pellet is needed. A striking instance of how this can now be done is provided by the recent work of T. A. Nuttall (3) working in the laboratories of the Council for Scientific and Industrial Research at Pretoria in a report on "Catalysts with Sub-surface Active Layers Prepared by Co-impregnation". Nuttall used the method of competitive adsorption, by taking solutions of chloroplatinic acid containing various concentrations of citric acid.

The citric acid or the citrate ion apparently adsorbs in competition with the chloroplatinate ion on the active centres on the alumina surface, so that the higher the citric acid concentration, the further the chloroplatinate ions can penetrate. In this way it proved possible to produce pellets in which the platinum after the reduction either was close to the surface, this being described as an "egg-shell" catalyst; or was confined to the centre of the pellet (an "egg-yolk" catalyst); or was located in an intermediate zone (an "egg-white" catalyst).

The effect of other variables, such as time of impregnation, drying and reduction conditions, on the concentration profile were also examined.

Thus, with improved procedures for controlling the location of the metal within catalyst particles, the way is now clear to experimental tests of theoretical models, and hence to a better appreciation of the role of diffusion within the pores of catalyst particles.

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### References

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- 3 T. A. Nuttall, CSIR Report CENG 182, published by the Council for Scientific and Industrial Research, Pretoria, South Africa