

## De-Oxo Plant Monitoring and Control

Recent years have seen a revolution in the production of nitrogen, particularly of the so called low grade variety containing 0.1 to 1% oxygen.

Whereas twenty years ago nitrogen was produced almost exclusively by the distillation of liquefied air, today pressure swing absorption plants (PSA) and membrane nitrogen generators are being used in growing numbers.

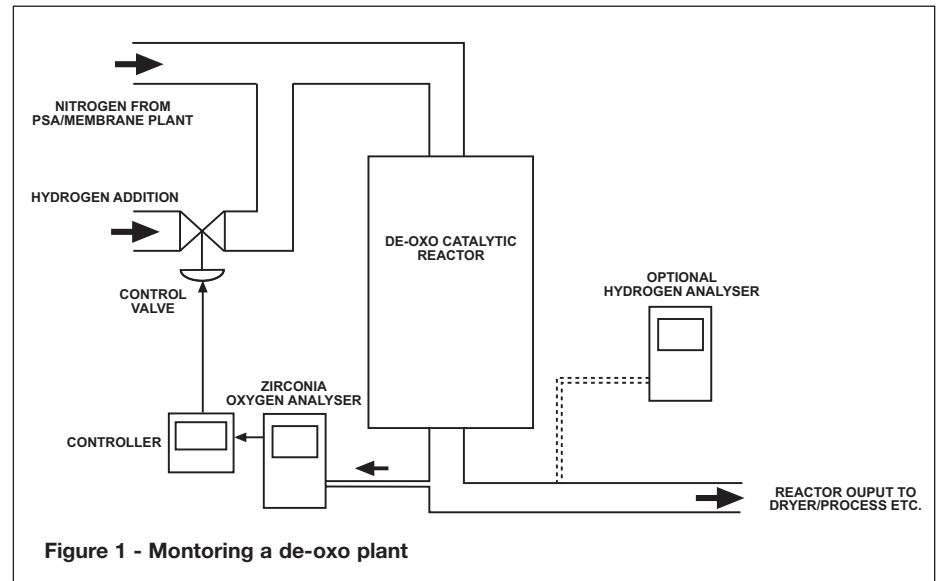
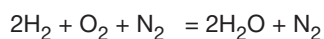


Figure 1 - Monitoring a de-oxo plant

These non cryogenic plants selectively remove oxygen from air to produce nitrogen (mainly) with a typical oxygen content of between 0.1 and 1%. Both systems, particularly PSA, can be designed to produce a much lower oxygen content, but it is frequently uneconomic to do so. As an alternative, de-oxo plants can be used to remove the final traces of oxygen from the products of these plants.

De-oxo plants work by adding hydrogen to the low grade nitrogen and react the resultant mixture by passing it through a column of catalytically active material. The hydrogen reacts with the residual oxygen to form water. The following chemical equation describes the reaction.



If necessary, the gas can then be passed through a dryer. For either technical or safety reasons many processes that use the gas produced by this system cannot tolerate any excess hydrogen in the product. The problem therefore is to add just sufficient hydrogen to react with the oxygen.

One of the most common approaches to the problem has been to monitor the oxygen concentration in the product from the nitrogen generator and its flow rate to the de-oxo reactor. These figures are used by a microprocessor system to compute the flow rate of hydrogen required to react with the oxygen.

The required flow rate is then set by means of an expensive mass-flow valve placed in the hydrogen supply line. As a safety check, the end products are monitored by a hydrogen analyser whose resolution is typically  $\pm 50\text{ppm}$  at best.

The method works but has two major disadvantages.

1. It is expensive; requiring precision analysers, flow meter, computers and mass flow control valves.
2. It results in the product containing noticeable amounts of hydrogen.

An alternative approach is to use an oxygen analyser produced by Hitech Instruments based on a zirconia oxygen sensor.

Zirconia oxygen sensors have a number of unique attributes two of which make them ideal for this application. Firstly, they can measure oxygen in concentrations ranging from 1 down to  $10^{-23}$  atmospheres; secondly, they can do this with a response rate measured in fractions of a second. They measure oxygen concentration in an analogous way to pH electrodes when measuring acidity.

In a way similar to a pH electrode being used to monitor the neutralising of an acid by an alkali, a zirconia oxygen sensor can be used to monitor a "neutralisation" of oxygen with hydrogen.

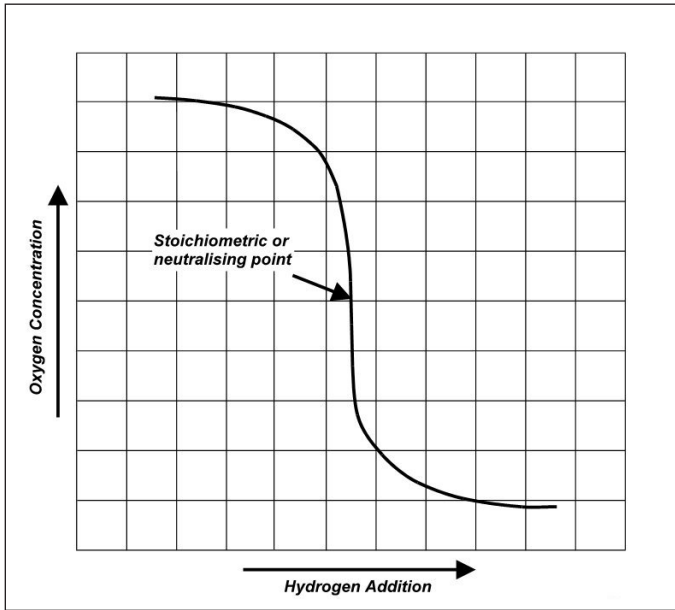


Figure 2 - Neutralisation curve

The graph in Figure 2 shows the classical neutralisation curve for such a process. The '*neutralisation*' point is where the reactants are in exactly the correct proportions to produce no excess of either. This is normally referred to as the '*stoichiometric*' point. At this point the concentrations of oxygen and hydrogen are due entirely to the dissociation of the water produced by the reaction and are in the order of parts per billion.

Fig. 1 (overleaf) shows the general arrangement for monitoring and controlling a de-oxo plant using a Hitech zirconia oxygen analyser.

Using this approach allows a lower cost conventional controller and control valve to be used as well as making the system fundamentally simpler. If required, an additional hydrogen analyser/monitor can be incorporated to monitor for a fault situation; e.g. the de-oxo reactor failing. Hitech can supply an oxygen analyser with this additional monitor incorporated.

**Note:** The Hitech G1010 oxygen analyser (H-type cell) and K1550 hydrogen analyser are used in this type of application.