High performance trace oxygen analysis in air separation plants

OXYMAT 64 is perfectly suited for this application

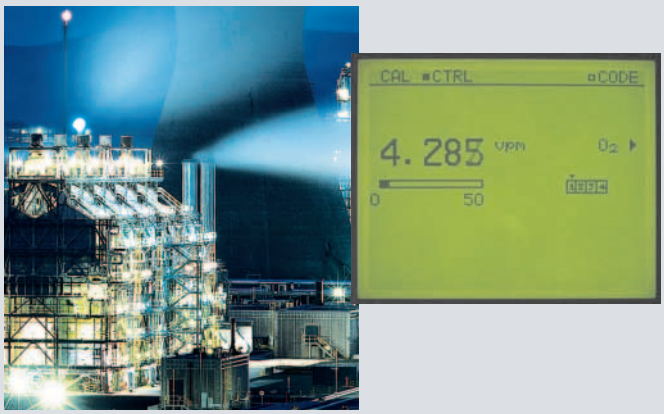
Case Study · November 2008

Air Separation

Large amounts of oxygen and nitrogen are required by many industries, e. g. chemical and petrochemical, metal, semiconductor and food industry, in machine construction and many others. Oxygen and nitrogen are, together with Argon, the main constituents of ambient air. They are obtained by air separation technologies in highly automated air separation plants. Two technologies are in use:

- Air separation at very low temperatures (cryogenic process) to liquefy the air and to produce the desired products by subsequent distillation based on differences in boiling points. This is by far the most common method.
- Air separation at higher pressure using adsorption effects based on differences in specific properties of the gases (pressure swing adsorption, PSA).

Cryogenic air separation processes consist of a similar series of steps independently whether large stand-alone plants or small and compact plants located directly at the consumers place are concerned. Variations reflect the desired product mix and individual priorities of the user.
The purity of oxygen produced ranges from 95 % upwards but is typically 99.95 % or better.



Process Analytics

SIEMENS

The Cryogenic Air Separation Process

Oxygen analysis

Process analyzers are used to control and optimize the processes and to ensure the product quality. Oxygen analysis in particular is of very high importance: oxygen in high percentages to ensure purity of the oxygen produced and trace oxygen concentrations to analyze the remaining traces in the pure nitrogen products.
Siemens Sensors and Communication is a leading supplier of Process Analytical Instrumentation and offers a wide scope of analyzers to various industries including the demanding air separation segment. OXYMAT 64 is especially designed to determine trace levels of oxygen in pure gases as it is required in air separation.

Process Steps

Cryogenic air separation processes use differences in boiling points of the components to separate air into the desired products oxygen, nitrogen and argon. These three gases represent about 99 % of dry ambient air. Numerous process configurations are utilized because of the demand of particular gas products and product mixes at various levels of purity. However, all cryogenic separation processes comprise filtering, compressing, purifying and rectification (fig. 1 and 2) of ambient air.

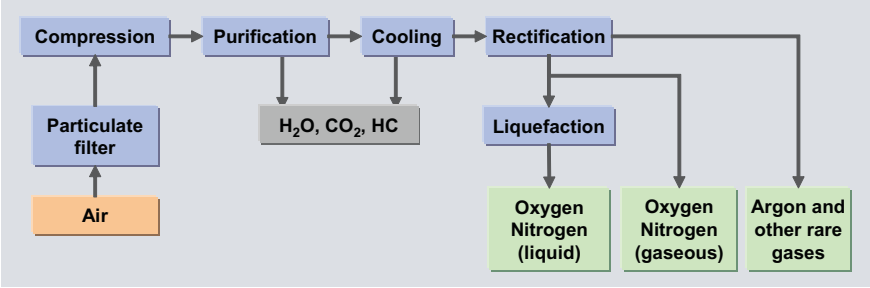


Fig. 2: Cryogenic air separation process steps

From filtering to cooling

Ambient air is sucked in through a filter and first compressed to approximately 6 bar. Then, by passing the air stream through a cooler and a molsieve, contaminants including water vapor, carbon dioxide (which would freeze in the process) and hydrocarbons are removed from the process stream. The compressed and purified air is then cooled in stages to very low temperature (-180 °C) through heat exchange and refrigeration processes in the main heat exchanger which is located in an insulated container called cold box. Heat exchange occurs in counter current against other streams such as product and waste nitrogen leaving the distillation columns. Products are warmed almost to ambient temperature while the process air is cooled close to liquefaction temperature. Final cooling is achieved by expanding the feed in an expansion engine.

Rectification (Separation)

Separation of air into its components is performed in a two-column rectification system comprising a high-pressure and a low-pressure column (fig. 1). A liquid oxygen-rich crude feed is produced as bottom product of the high pressure column: in a counter-current system a gas stream rises upwards the column while a liquid mixture flows down. High boiling liquid oxygen is formed from the rising gas stream by condensation while, from the liquid, lower boiling nitrogen is evaporated. Thus, gaseous nitrogen is formed at the top of the column while liquid oxygen is produced at the bottom. This process runs continuously and is called rectification. Pure nitrogen is eventually taken off the overhead of the column.
The oxygen-rich crude feed from the bottom of the high-pressure column becomes feed to the low-pressure column for final separation into pure oxygen (at the bottom) and a oxygen containing nitrogen fraction that is withdrawn from the top of the column. Oxygen is withdrawn from the bottom as product. Achievable purities depend on the amount of ambient air fed to the process and the number of separation trays of the columns.

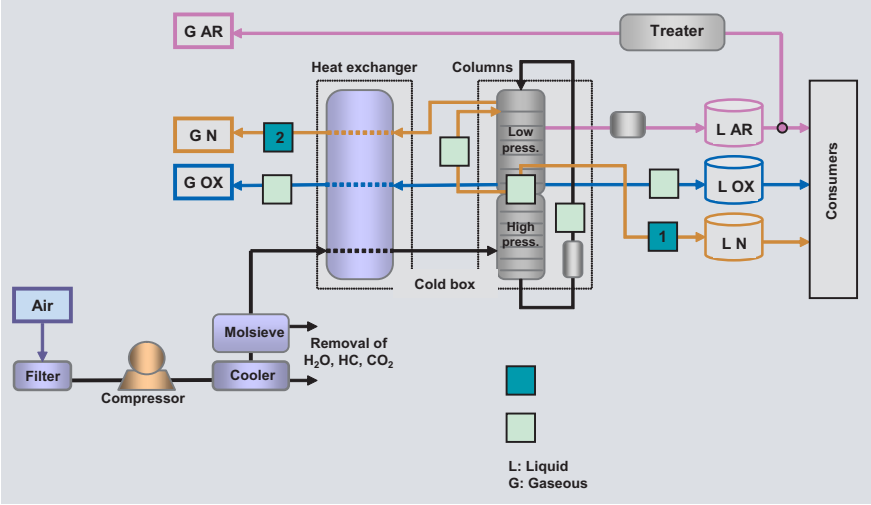


Fig. 1: Cryogenic air separation process: oxygen analysis locations

Trace Analysis of Oxygen by Using OXYMAT 64

Argon is enriched in the middle part of the low pressure column. It can be withdrawn from there and processed to pure argon in additional concentrating steps.

Process gas analyzers

Reducing energy consumption and ensuring product quality are dominating goals in air separation plant operation. Process gas analyzers are used at various locations throughout the process to provide the required concentration data of the process streams with oxygen being the most important component. The respective measuring locations are indicated in fig. 1.
Siemens Process Analytics holds a very strong market position in oxygen gas analysis with three analyzer models: OXYMAT 6/61 for analysis up to highest concentration ranges (98 to 100 %) and the new OXYMAT 64 for trace analysis down to the vpm range.

Trace analysis

Trace oxygen analysis is required at the measuring points 1 and 2 (fig. 1) to ensure the purity of the products liquid nitrogen and gaseous nitrogen. But also higher concentrations, which may occur during plant start-up, can be measured using the analyzers automatic measuring range switchover functionality.

Technical data of OXYMAT 64	
Smallest measuring range Largest measuring range	0 ... 10 vpm 0 ... 100 %
Number of measuring ranges	4
Detection limit	100 vpm (1 % of measuring range))
Linearity	< 2 % of measuting range
Drift (Messwert)	< 2 % of measuring range / month
Pressure: Low pressure version High presure version (for gas bottle filling)	 Ambient 2000 ... 6000 hPa
Sample gas temperature	0 ... 50 °C
Response time (T90): Low pressure version High pressure version	< 3 s 10 ... 30 s
Communication Interfaces	RS 232, RS 485 PROFIBUS DP/PA (option)

Table 1: OXYMAT 64, technical data

OXYMAT 64

The new developed OXYMAT 64 analyzer (fig. 3) belongs to the well known and proved Series 6 of Siemens continuous extractive gas analyzers. Its preferred application area is the determination of very low levels of oxygen in pure gases and thus, OXYMAT 64 is a key tool for high performance trace oxygen analysis in air separation plants.

Zirconium dioxide technology

OXYMAT 64 uses ZrO_2 technology with a tubular sensor that is heated continuously to 650 °C. While the sample gas flows through the sensor, the outer side of the sensor is exposed to ambient air which acts as reference electrode (fig. 4). The different oxygen concentration across both sides of the sensor results in different partial pressures causing ion movement and creating a potential difference between both electrodes. This potential difference is proportional to the oxygen concentration in the sample gas.

Fig. 3: OXYMAT 64 trace oxygen analyzer

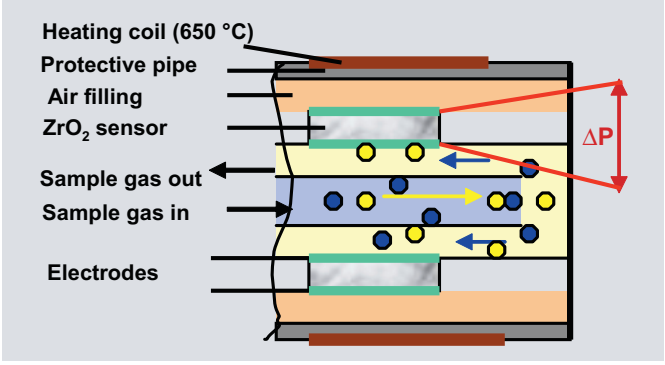


Fig. 4: OXYMAT 64, sensor principle

Siemens OXYMAT 64 Benefits

Key features

- Especially designed for trace oxygen analysis with smallest measuring range 0 ... 10 vpm and detection limit 100 ppb O_2
- Two pressure versions available for plant control and bottle filling application
- Four measuring ranges with automatic switch-over
- Designed for highly reliable and accurate measuring results with linearity deviation < 2 % of measuring span and drift < 2 % of measuring span / month
- Choice of versions with catalytic inactive or catalytic active sensor with different reactivity to combustibles present in the sample gas
- Menu guided operation according to NAMUR recommendation
- Cost efficient readjustment with ambient air
- Open interface architecture providing RS 485, RS 232, PROFIBUS DP/PA (Option)
- Two applications with one analyzer: Plant control (low pressure) and bottle filling (high pressure)
- Easy integration into plant communication networks because of versatile interfacing options

The benefits by using OXYMAT 64 for trace oxygen analysis arise both from the technical performance of the analyzer and the strength of Siemens Process Analytics in providing complete analysis solutions and excellent and versatile support worldwide.

Analyzer

- Very reliable and accurate measuring results because of the very high signal strength at trace concentrations (principle of ZrO_2 technology)
- Besides traces, higher O_2 concentrations can be measured with the same analyzer, through automatic range switching, e. g. during plant start-up
- Low training efforts and little operating errors because of self-explaining menuguided operation
- Optimal measuring results due to very low cross sensitivity to combustibles (special sensor technology)

Complete analysis solutions

In addition to deliver an excellent trace oxygen analyzer to either end user or plant manufacturers, Siemens Process Analytics is well prepared and experienced to equip air separation plants with complete analysis solutions ranging from planning and engineering to installation, commissioning and maintenance. We offer an integrated design covering all steps from sampling point and sample preparation up to complete analyzer cabinets or for installation in analyzer shelters. This includes also signal processing and communications to the control room and process control system.

Service and Support

Siemens Process Analytics is your competent and reliable partner world wide with a broad portfolio of Service, Support and Consulting (fig. 5). Our resources for that are:

Expertise

As a manufacturer of a broad variety of analyzers, Siemens Process Analytics is very much experienced in engineering and manufacturing of analytical systems and analyzer houses including communication networks, well trained in service and maintenance and familiar with many industrial processes and industries. Thus, Siemens Process Analytics owns a unique blend of overall analytical expertise and experience.

Global presence

With the strategically located centers of competence in Germany, USA, Singapore, Dubai and Shanghai, Siemens Process Analytics is globally present and acquainted with all respective local and regional requirements, codes and standards. All centers are networked together.

Fig. 5: Portf olio of services

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