SERVOMEX

Servomex Total Solution for Gas Analysis on Cryogenic Air Separation Plants

Application

Servomex provides gas analysers which have been specifically designed to meet the process control, quality assurance and safety monitoring requirements of air separation plants.

Background

Dry air contains approximately 78% nitrogen, 21% oxygen, and 1% argon plus low concentrations of noble gases, carbon dioxide, hydrocarbons and other impurities. Nitrogen, oxygen and argon are used by industry in large quantities and hence termed industrial gases. To support this need processes have been developed to produce these gases through the separation of air.

An air separation plant divides atmospheric air into the three pure gaseous components of nitrogen, oxygen and argon. Further separation may be performed on some plants to produce other gases such as neon, krypton and xenon. Other gas components of atmospheric air, such as water vapour, carbon dioxide and hydrocarbons must be removed to ensure safety, efficient plant operation and product quality.

There are different types of air separation plants but those based on cryogenic air separation are the most significant in terms of production value and volume. Cryogenic air separation presents the most opportunity for gas analysis and is now discussed in more detail.

SERVOMEX

Cryogenic Air Separation

Large air separation plants are based upon the cryogenic fractionation of air into its pure components. This process is based on the principle that the desired products have different boiling points and thus can be separated out from liquefied air. Due to differing user requirements there are a number of variations in the air separation cycles which are used to make industrial gas products. However all cryogenic air separation processes consist of a similar series of steps: see figure 1.

Air compression and filtering:

The ambient air feed is compressed by a multiple stage compressor with intercoolers to a pressure of about 6 bara. Dust is removed by a filter located at the inlet to the compressor.

Air cooling and purification:

The compressed air is then cooled to close-to-ambient temperature by passing through a direct contact water cooler. Chilling of the cooling water is done in an evaporation cooler using dry nitrogen waste gas from the process. Much of the water vapour in the incoming air is condensed and removed as the air passes through the compressor and cooler. Most of the remaining water vapour, carbon dioxide and hydrocarbons are removed by molecular sieve absorbers.

Refrigeration/Liquefaction:

The purified compressed air feed is then cooled by heat exchangers and refrigeration processes contained in the "cold box" where it reaches cryogenic temperature of around -180˚C. Final cooling is done by expanding the feed in an expansion engine. The resulting mixture of liquid and vapour air is separated and fed to the separation column.

Separation:

This separation column consists of a high pressure and low pressure column where air is separated into its pure components. The mixture of liquid and vapour air from the refrigeration/liquefaction stage is fed into the high pressure column. This mixture separates into nitrogen at the top and oxygen enriched air at the bottom.

This oxygen enriched air is fed into the low pressure column where it is further purified. Pure nitrogen is finally taken off the top of the high and low pressure columns and pure oxygen is withdrawn from the bottom of the low pressure column.

Argon is enriched in the middle part of the low pressure column, the so-called argon belly. It can be withdrawn from there and processed to pure argon in additional concentrating steps as follows.

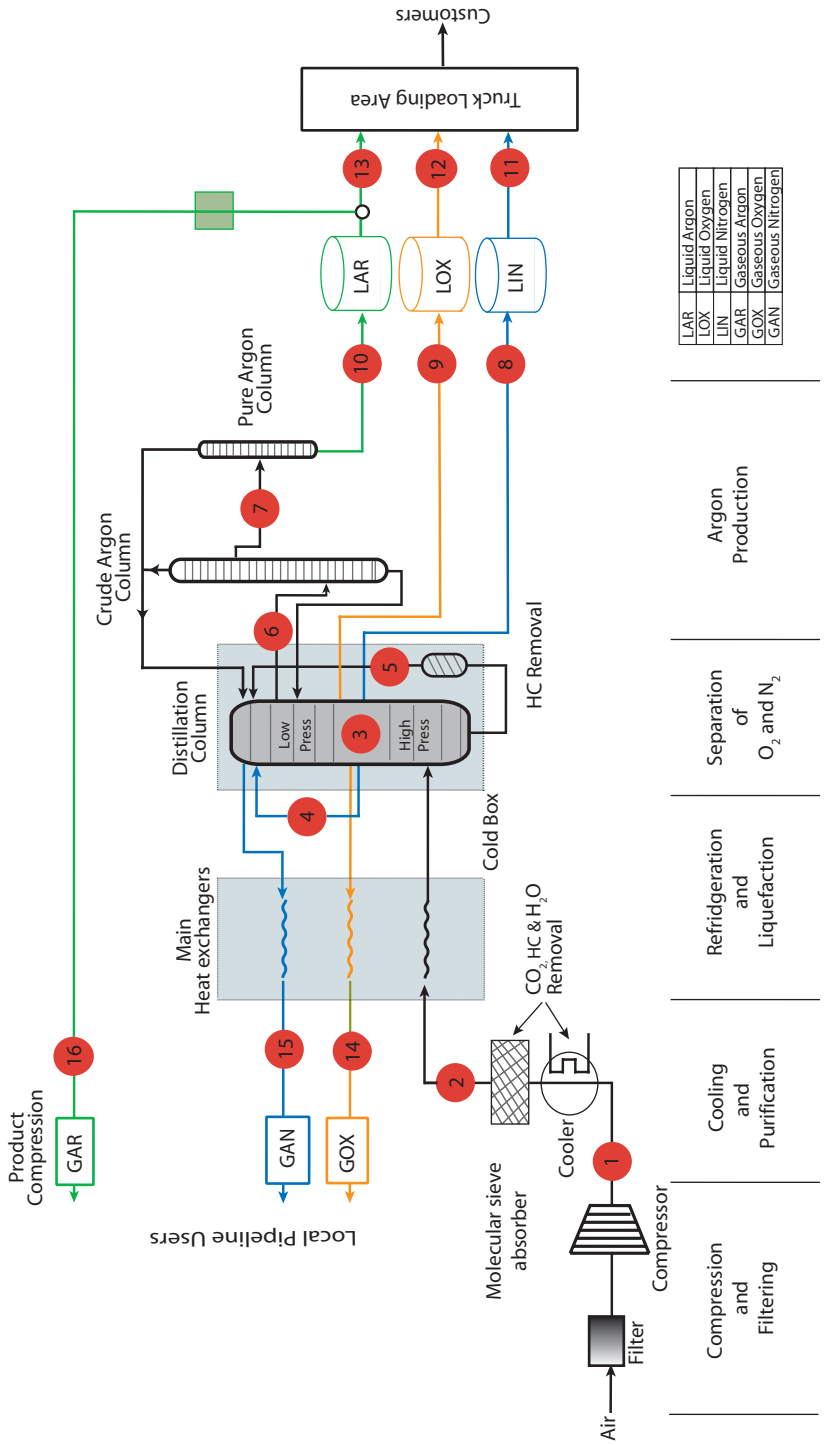
Pure argon production:

In the past argon was produced by feeding the stream from the argon belly in the low pressure column to the crude argon column. Then the product from this is passed through a catalytic converter to remove the remaining oxygen before any nitrogen is removed in the pure argon column.

The modern process is to pass the stream from the low pressure column into the crude argon column for further separation. The remaining oxygen in this gas stream is completely removed in this packed column. The oxygen free argon stream is then fed into the pure argon column where the remaining nitrogen is removed by separation and the pure argon is liquefied.

SERVOMEX

Figure 1 Typical Air Separation Process



SERVOMEX

Table 1: Typical gas analysis (refer to ASU flow schematic Figure 1)

Analysis position	Sample stream	Measuring component	Typical range	Main purpose	Suitable Servomex analysers
1	Process air before molecular sieve	THC Impurities	0-10/100ppm 0-10/100ppm	Safety	FID (K1000) Chroma (K4000)
2	Feed to high pressure column	CO_2 THC	0-10ppm 0-10/100ppm	Safety	MultiEact,4100 FID (K1000)
3	Low pressure column liquid phase	O_2 CO_2 N_2O THC	98-100% 0-10ppm 0-20ppm 0-100/1000ppm	Control Safety	MultiExact,4100 MultiExact,4100 MultiExact,4100 FID (K1000)
4	Distillation column process stream	O_2	0-20%	Control	MultiExact,4100
5	Feed to low pressure column	O_2	0-50%	Control	MultiExact,4100
6	Crude argon column feed	O_2 N_2 Ar	80-100% 0-2000ppm 0-20%	Control	MultiExact,4100 Chroma (K4000) K1550
7*	Feed to pure argon column	O_2 N_2 Ar	0-1/10ppm 0-5000ppm 80-100%	Control	310E/MultiExact,4100 Plasma (K2001) K1550
8, 11	Liquid nitrogen before and after storage	O_2 Impurities	0-1/10ppm 0-10ppm	Control/Quality	310E/MultiExact,4100 Chroma (K4000)
9, 12	Liquid oxygen before and after storage	O_2 Impurities	98-100% 0-10ppm	Control/Quality	MultiExact,4100 Chroma (K4000)
10, 13	Liquid argon before and after storage	O_2 N_2 Impurities Ar	0-1/10ppm 0-10ppm 0-10ppm 80-100%	Control/Quality	310E/MultiExact,4100 Plasma (K2001) Chroma (K4000) K1550
14	Gaseous oxygen	O_2 CO_2 THC Impurities	98-100% 0-10ppm 0-10ppm 0-10ppm	Quality	MultiExact,4100 MultiExact,4100 FID (K1000) Chroma (K4000)
15	Gaseous nitrogen	O_2 Impurities	0-1/10ppm 0-10ppm	Quality	310E/5400,4100 Chroma (K4000)
16	Gaseous argon	O_2 Impurities Ar	0-1/10ppm 0-10ppm 80-100%	Quality	310E/MultiExact,4100 Chroma (K4000) K1550

* Only applies to modern processes using packed crude argon columns

SERVOMEX

Analysis Requirements

Minimising production costs whilst maintaining product quality and safety is a major challenge for air separation units. Throughout the process gas analysers are employed to provide continuous and reliable analysis to ensure that this happens. Figure 1 is a diagram of a typical cryogenic air separation plant. On this are shown the usual analysis points and Table 1 relates these points to the required analysis.

Oxygen analysis % levels

Oxygen analysis is required to control the process and to verify the quality of the oxygen product. The SERVOPRO MultiExact (5400) and SERVOPRO 4100 are very suitable for this because they use the inherently linear paramagnetic oxygen cell with pressure compensation. This enables them to measure up to 100% oxygen without the need for special reference gases with an accuracy of 0.01% when the MultiExact is used, or 0.02% when the 4100 is used.

Oxygen analysis ppm levels

At the other extreme oxygen analysis typically in the range 0 to 10 ppm is also required and the zirconia technology employed by the SERVOPRO MultiExact (5400) and SERVOPRO 4100 is ideal for this. The unique zirconia sensor employs an inhibited catalytic design which means that it is not adversely affected by the presence of trace combustible gas in the sample. This together with its fast speed of response makes it ideal for monitoring the quality of the nitrogen and argon product streams where oxygen is an unwanted impurity.

Oxygen analysis ppb levels

For verifying the quality of the argon and nitrogen product streams prior to supply to the customer, an oxygen analysis at sub ppm levels is frequently required. This can readily be done using the Delta F DF-310E analyser using its unique non depleting coulometric sensor technology. The ability to analyse ppb levels in oxygen make it ideal for quality analysis of the end product.

Carbon dioxide analysis

For safety and quality reasons carbon dioxide must be removed from the ambient air feed and analysis is required to verify this. Carbon dioxide analysis is also needed to ensure that the final products meet the required specifications. The SERVOPRO MultiExact (5400) and SERVOPRO 4100 use Gas Filter Correlation (GFC) technology for this which ensures a specific, sensitive and stable measurement making it especially suitable for this analysis.

Nitrous oxide analysis

Nitrous oxide can accumulate in the low pressure column which may cause safety issues and also contaminate the product. To check for this analysis is required from this region. Once again the SERVOPRO MultiExact (5400) and SERVOPRO 4100 use the reliable GFC technology for this.

SERVOMEX

Total hydrocarbon (THC) analysis

Total hydrocarbons are monitored in the air inlet feedstock to ensure that the process scrubbers can safely handle the concentrations present and for safety reasons they are also analysed in the low pressure column. Additionally for quality purposes total hydrocarbons are monitored in the product streams. These measurements are readily achieved using the SERVOPRO Chroma (K4000) or SERVOPRO Plasma (K2001) gas analysers.

Trace impurities analysis

Trace impurities are analysed in the final product streams to ensure that they are within the specified limits. The SERVOPRO Chroma (K4000) is ideal for this analysis because it can detect a wide range of impurities down to ppb levels.

Argon analysis

To optimise Argon production and check the product quality it is necessary to analyse it. This is done using a K1550 Thermal Conductivity Detector which is optimised to measure between 30 and 100% Argon in the product streams.

Nitrogen analysis

Nitrogen is analysed using the SERVOPRO Chroma (K4000) to enable tight process control of argon production resulting in improved yield. Nitrogen is also analysed in certain product streams to ensure that it meets the required specification. The SERVOPRO Plasma (K2001) is ideal for this due to its ability to measure concentrations below 1ppm.

Water vapour analysis

Too much water vapour can lead to ice formation in the heat exchanger. This may cause a blockage, leading to build up of pressure and unsafe operating conditions. Thus it is important to monitor water vapour to ensure that the concentrations do not exceed safety limits. For quality purposes water vapour may also be checked in the final product streams.

Servomex Analysers

Servomex offers a wide range of analysers to provide a total gas analysis solution for air separation units. A brief summary of these and the analysis follows.

SERVOPRO MultiExact (5400)

Various combinations of the above analyses can be done in one SERVOPRO MultiExact (5400) analyser which can analyse up to 2 components at the same time.

SERVOPRO MultiExact (5400) key features:

- Measures up to two gas streams simultaneously
- Unparalleled operational performance from enhanced sensor technologies
- Low cost of ownership. Non depleting stable sensors, no need for frequent calibration
- Comprehensive digital communications. Profibus, Modbus, Ethernet

SERVOMEX

DELTA F DF-310E

The DF-310E Oxygen Analyser utilises the renowned Delta F proprietary non depleting coulometric sensor technology, with the advantages of a 5 year sensor warranty, span calibrations required only once per annum, and no false low readings. The sensor arrives factory calibrated, and does not require calibration as part of commissioning.

DELTA F DF-310E key features:

- Suitable for the measurement of ppb, ppm and % level oxygen measurements
- Rugged compact analyser available in bench mount, panel mount, and rack mount versions
- Low cost of ownership. No requirement for frequent span calibration and periodic sensor replacement
- Ideal for reliable, precise QA measurements. No false low readings due to the sensor depletion experienced with other electrochemical sensor technologies

SERVOPRO Chroma (K4000)

The SERVOPRO Chroma (K4000) is a multicomponent gas chromatograph which uses plasma technology to measure trace levels of specific impurities found in the O_2, N_2 and Ar product streams. The SERVOPRO Chroma (K4000) is also used to measure trace N_2 in crude Ar in order to increase argon yield by as much as 5%.

Chroma key features:

- Trace gas measurements ppb/ppm/%
- Analyses O_2, N_2, Ar, CH_4, NMHC, CO, CO_2,H_2 impurities in pure Ar, O_2 and N_2
- Stand alone system, no need for third party software
- Compact rack mount 4U configuration
- Electronic platform for easy operation
- Internet enabled for remote control and monitoring

SERVOMEX

SERVOPRO Plasma (K2001)

The SERVOPRO Plasma (K2001) is a Plasma Emission Detector based analyser which designed to continuously measure N_2 in pure Ar. It can also be configured to measure N_2 impurities in pure He and Ar in gas bottling and specialty gas laboratory applications.

Plasma key features:

- Sensitive, stable plasma emission detector
- Solid state mass flow transducer controller
- Standard factory set 3 measuring ranges
- Automatic range change for optimal output resolution
- Digital communications for remote monitoring

SERVOPRO FID (K1000)

The SERVOPRO FID (K1000) is a Flame Ionisation Detector based analyser designed to ensure safe operation of cryogenic air separation units by checking that the concentration of total hydrocarbons (THC's) is below flammable limits as well as provide quality control in pure O_2, N_2, Ar, He and Air.

FID key features:

- Reliable, stable Flame Ionisation Detector
- Standard factory set 3 measuring ranges with over range
- Automatic range change for optimal output resolution
- Flame out alarm and fuel shut off valve
- Digital communications

SERVOPRO 4100

The SERVOPRO 4100 is a cost effective multicomponent analyser, which may be used when more than two components are required to be analysed. It is used for the analysis of % O_2, trace O_2, trace CO_2, trace CO, trace N_2O and trace H_2O using an external moisture transducer.

K1550

The K1550 is a thermal conductivity analyser for analysing % levels of Ar in air separation units.

SERVOMEX

Summary

Whether the purpose is safety, product quality or process control, Servomex is able to offer the total gas analysis solution for air separation units.

Service & Support

For new installations and replacement of older Servomex and competitor products, we will work with you to develop a bespoke service and support package, ensuring full measurement availability and plant operation within your timescales and budget.

SERVO**SPARES**

To ensure the integrity and optimum performance of your Servomex product, we recommend fitting only factory authorised spare parts. This is particularly important for all hazardous area certified products.

SERVO**SURE**

Ensure your Servomex analyser is properly commissioned and delivers optimum performance with a maintenance contract, service programme and extended warranty.

SERVO**TECH**

Make the most of your Servomex gas analyser by attending a training course at one of our training centres in Europe, USA or Asia or on your own site.

SERVO**HELP**

Whether you have a simple question or complex process challenge, our local offices and global support network are here to help you.

SERVOMEX

BUSINESS CENTRES

EUROPE (Europe and Africa)
Tel: +31 (0)79 330 1581
Fax: +31 (0)79 342 0819
Toll Free : 00800 7378 6639

USA & CANADA
Tel: +1 281 295 5800
Fax: +1 281 295 5899
Toll Free: 1 800 862 0200

LATIN AMERICA/MEXICO
Tel: +55 11 5188 8166
Fax: +55 11 5188 8169

ASIA PACIFIC
Tel: +86 (0)21 6489 7570
Fax: +86 (0)21 6442 6498

INDIA AND MIDDLE EAST
Tel: +91 22 67100359/60
Fax: +91 22 66778896

TECHNICAL CENTRE

Servomex Group Limited
Crowborough
East Sussex, UK
TN6 3FB
Tel: +44 (0)1892 652181
Fax: +44 (0)1892 662253

SYSTEMS ENGINEERING CENTRES

Crowborough, UK	Tel: +44 (0)1892 652181
Houston, USA	Tel: +1 281 295 5800
Shanghai, China	Tel: +86 (0)21 6489 7570
Mumbai, India	Tel: +91 22 67100359/60

SERVOMEX
A MEASURABLE ADVANTAGE

www.servomex.com

HUMMINGBIRD
SENSING TECHNOLOGY

www.hummingbirdsensing.com

Servomex has a policy of constant product improvement and reserves the right to change specifications without notice.
© Servomex Group Limited. 2011. A Spectris company. All rights reserved.

AB15 Rev.0 04/11 E