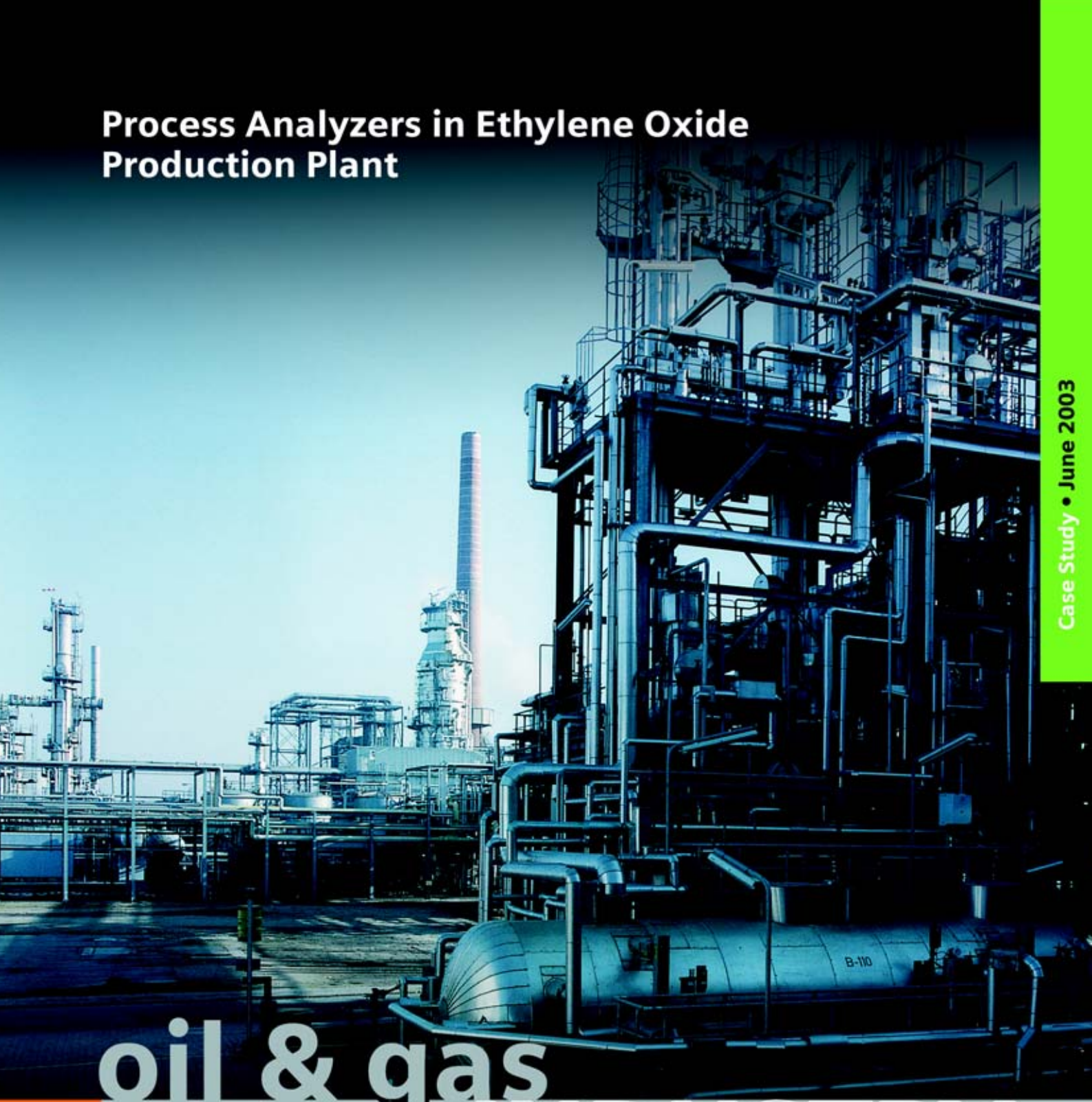
Process Analyzers in Ethylene Oxide Production Plant



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Production of Ethylene Oxide

Ethylene oxide, C_2H_4O, is a colorless, flammable gas or liquid. Because of its molecular structure ethylene oxide is one of the most versatile chemical intermediates. Industrial production started in 1925 using the chlorohydrin process and was improved in 1931 by introducing the much more economic direct catalytic oxidation method. Currently, almost all ethylene oxide production plants are based on the direct oxidation process with air or oxygen.

Ethylene oxide itself is an excellent disinfectant, sterilizing agent, and fumigant, when it is used as a non-explosive mixture with N_2 or CO_2. However, most of it is converted into other products such as fibers, foils, bottles, plasticizers, solvents, antifreezes, cosmetics etc.

Production of Ethylene Oxide

Production process is based on a catalytic reaction between ethylene and oxygen using silver as catalyst. No other metal until now can compete with silver, which, in practice, is deposited on various kinds of porous support materials. The exact composition and structure of the support material do strongly influence to process and product quality. It is considered as know how of the producers and therefore kept secret.

Several, but similar, technologies exist for ethylene oxide production. Differences depend mainly on whether air or pure oxygen is used for oxidation. Production steps for the oxygen-based process are described below (text box).

Ethylene oxide can cause heavy accidents because it may explode and is both highly flammable and reactive. Furthermore it is toxic. Therefore a great number of safety measures must be applied in a plant to prevent

- Leakage of liquid or gaseous ethylene oxide,
- Entry of air or oxygen into ethylene oxide containers,
- Ignition sources, and
- Overheating of ethylene oxide.

Fig. 1 Ethylene Oxide production plant

Steps of oxygen-based ethylene oxide processing

- Feed of ethylene and oxygen into the reactor and oxidation of ethylene by passing through the reactor (bundles of tubes packed with the catalyst material) at 200 to 300 °C. Together with ethylene oxide, CO_2, H_2O, and heat is generated as well.
- Cooling of the gas that leaves the reactor by means of steam generation or direct heating of the gas inlet of the reactor.
- Removal of ethylene oxide and CO_2 from the gas by scrubbing with first water and second an aqueous potassium carbonate solution. The ethylene oxide dissolves in the solution.
- Desorption of ethylene oxide from the solution in the desorber and stripping of its low-boiling components.
- Final distillation with separation into water and ethylene oxide.

The process runs continuously using a recycled gas stream through the reactors driven by compressors, and reloaded before entering the reactor again.

Process Analysis Instrumentation of Ethylene Oxide Plants

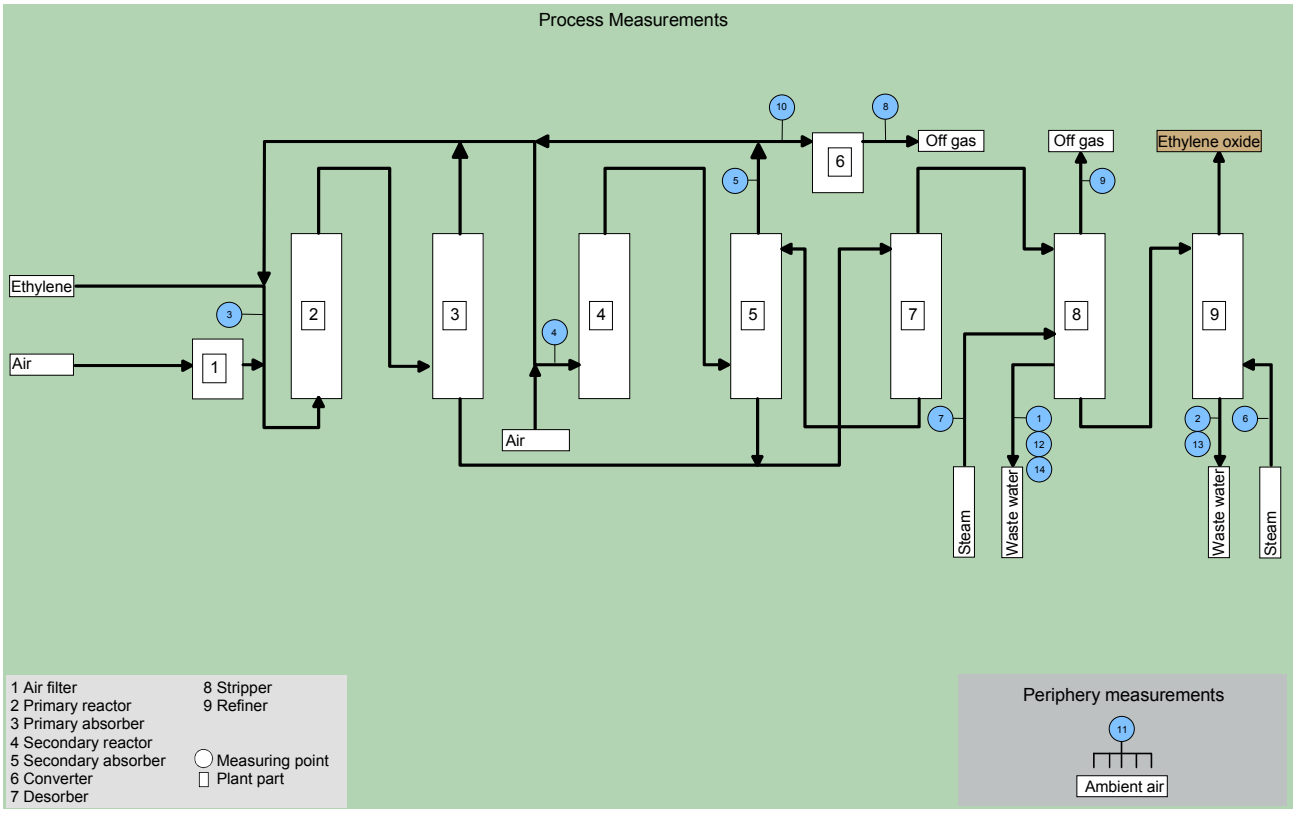

Fig. 2 Process flow chart with measuring locationsg

Process Analyzers

Process analyzers are a very important part of an ethylene oxide plant field instrumentation.

Measuring tasks can be grouped into

- Process control and optimization
- Product quality control
- Personnel and plant safety control
- Environmental compliance control.

To perform these analysis tasks a high number of different analyzers is installed in an ethylene oxide plant ranking from simple liquid analyzers up to process gas chromatographs and mass spectrometers. Measurements include raw material and final products, off gases and waste water, ambient air and, of course, the process gas at various locations in the process.

Process analyzers play an important part within the field instrumentation of an ethylene oxide plant. Users demand a >99 % device availability and expect respective measures built into the analyzers, such as status signal capability etc. The users, on their side, are also prepared to contribute to that demand by providing adequate conditions for installation and operation of the analyzers.

Oxygen measurement

The determination of the oxygen content of the process gas up- and downstream of the reactor (ca. 8 % resp. 6 % O_2, see measuring points 3 and 5 in fig. 2) is of particular importance. Plant efficiency increases proportionally to the oxygen content of the process gas and therefore, for economical reasons, the plant shall be run at oxygen contents as high as possible, close to the explosion limit, but still reliably distant from that. The distance to the explosion limit can be kept so smaller and the plant efficiency so higher the more accurate and reliable the oxygen measurement is performed. 1 % higher oxygen content in the process gas will easily correspond with an additional production rate of several hundreds of tons ethylene oxide per year. Therefore, the use of the best available oxygen measuring technology, as offered by the Siemens OXYMAT analyzer series, is a very economic decision.

Usually the oxygen measurement is performed redundantly with, for instance, three separate analyzers installed at one measuring point.

In case of a fault, a „2 out of 3" signal evaluation principle is applied and processed for appropriate safety measures.

Gas Chromatographs and Mass Spectrometers

are both indispensable in a modern ethylene oxide plant. Their application depend on criteria such as suitability for certain components, selectivity, response time characteristic, or investment and operation costs. Both of them have preferable application fields. Latest developments, e.g. the Siemens MAXUM edition II Gas Chromatograph or the Siemens Quantra Mass Spectrometer perhaps will cause some alterations of these application fields in the future.

Siemens Analyzers and Analyzer Systems

Siemens Process Analytics is, since a long time, well known for its wide spectrum of gas chromatographs, continuous gas analyzers and liquid analyzers, which are all highly qualified and proven for use in a ethylene oxide plant, also in case of plant revamping projects. The product line has been extended during the past years and comprises to day e.g. also the Quantra Mass Spectrometer which offers remarkable technical features for use in an ethylene oxide plant.

Besides the analyzer product business, Siemens Process Analytics is one of the worlds leading system supplier of analyzer systems with manufacturing facilities in Europe, America and Asia, with a wide application know how including in-depth experience in supplying analyzer systems to ethylene oxide plants.

Features OXYMAT 6	User Benefits
Simple and robust design without moving parts	High operating reliability, availability, and service life; very low maintenance and spare part requirements
Strictly linear measuring principle	High precision and flexibility
Very short signal response time	T_{90} time <2 s provides optimum reaction to process variations
Measuring principle allows differential measurement of two different gas streams in one analyzer with one bench No electronic zero surpression required	Very small measuring ranges for high (absolute) concentrations and thus very high measuring precision
Minimum drift (0.5 % of span in 3 months)	Very high measuring precision Very little needs for recalibration
Remote control by SIPROM GA software tool Interface to PROFIBUS PA (option)	Easy integration into automated systems
Available in one housing together with an ULTRAMAT 6 NDIR analyzer	Very economic operation

Table 1 OXYMAT 6, analyzer features and user benefits

Features MAXUM edition II	User Benefits
Single or dual oven design, with 1 or 2 channels each, for use in areas with or without explosion hazards	Optimum customization to actual analysis task Extremely cost efficient operation
Retrofit possible to include second oven later	Low-cost expansion capability
Wide range of detectors available, including highly sensitive HID (Helium detector) for trace analysis in high purity gases	Most versatile for optimum customization to analysis task
Valveless Live Column Switching technology	Increased analysis capabilities Lower maintenance requirements
Parallel Chromatography: One complex application is split into a number of simple and parallel running applications	Dramatically reduced cycle times Simplified maintenance
Electronic pressure controller	No needle valves required
TCP/IP communication via Ethernet	Compatible to many networks
High number of data processing methods available, e.g. area normalization, internal and external standard, and 100 % method	Extremely precise analysis results

Table 2 MAXUM, analyzer features and user benefits

Some of the Siemens analyzers with their technical features and corresponding user benefits are described in the following.

Fig. 3 Series 6 Gas Analyzer in field housing

The **OXYMAT 6** is a gas analyzer that operates according to the paramagnetic principle and is designed for high-precision measurements of oxygen concentrations in gases. The pulsating magnetic field creates minute flow pulses detected by the Siemens micro-flow sensor and converted into the measuring signal. Thus, the OXYMAT 6 does not contain any moving parts. The sample stream gas also does not come into contact with the microflow sensor, which ensures an extremely long life time and high operating stability. A good example for that is the independence from humidity which may be present in the samplegas and could cover the surface of mirrors (with negative impact to the measuring accuracy) that are used with other oxygen measuring principles.

The **ULTRAMAT 6** is a gas analyzer that operates according to the non-dispersive infrared principle (NDIR). It is designed to perform highly selective concentration measurements of infrared-sensitive gases. The ULTRAMAT 6 uses the two-beam alternating light principle with a measurement and comparison cell, dual-layer detector, and optical coupler.

This optical bench design produces an extremely narrow absorption curve minimizing the influence of overlapping spectra. Thus an unparalleled analytical precision is obtained. Cross sensitivities (caused by physical facts) are minimized by using the gas filled beam splitter, the dual layer detector with optical coupler (optical extension of the detector chamber) and additional filter.

Features ULTRAMAT 6	User Benefits
Dual-layer detector with variable optical path length setting (Optocoupler)	Maximum selectivity and thus measuring precision. Can be optimized for actual analysis
Detector uses microflow sensor with no moving parts to generate the measuring signal	No microphony: Very low signal noise High measuring accuracy
Extremely stable mechanical design Electronic and physical parts separated gas tight in one robust IP 65 housing	Very high operating reliability and life time
Easy cleaning of gas cell (on site possible)	Minimum maintenance costs
Remote control by SIPROM GA software tool Interface to PROFIBUS PA (Option)	Easily integrated in automated systems

Table 3 ULTRAMAT 6, analyzer features and user benefits

Features Quantra	User Benefits
Very high Mass Resolution (20 000 at 100 amu)	No interferences of components of similar mass
Exact mass determination Mass Accuracy ± 0,0004 amu	Correct identification of components Identification of unknown components
"Ion Ejection" application tool	Discharge of high concentrated components for improved detection capability
Robust design of measurement cell, filament, and vacuum system	Suitable for operation in harsh process environment
No moving parts contained	Long service life, very low maintenance needs
Ion pump	Cell vacuum 10^{-10} Torr No mechanical vacuum pumps required

Table 4 Quantra, analyzer features and user benefits

The **MAXUM edition II Process Gas Chromatograph** (Fig. 4) combines the outstanding analytical features of the Siemens PGC 302 Process Gas Chromatograph with the *Parallel Chromatography* (separation of complex analyzing tasks into several more simple steps) and the ethernet communication technology of the Advance MAXUM gas chromatograph of Applied Automation in one new device. Thus the MAXUM edition II is a gas chromatograph of outstanding performance and very high flexibility. The single or double oven concept, the wide selection of columns and detectors including the valveless column switching technology, and the modern ethernet communication capability allows a highly selective and sensitive analysis of most different process components.

Fig. 4 MAXUM edition II Gas Chromatograph

The **Quantra Mass Spectrometer** by Siemens (Fig. 5) is the first high resolution mass spectrometer world wide that is capable to perform online applications.

Quantra is well suited for a number of process applications such as trace gas analysis and ambient air monitoring in the chemical industry, or residual gas analysis in the semiconductor industry. The ICR (Ion Cyclotron Resonance) technology provides an unmatched mass resolution which allows to reliably identify molecules of almost the same mass.

Quantra was specifically designed for use in harsh process environments with easy operating procedures and very low maintenance requirements.

Fig. 5 Quantra Mass Spectrometer

With **SIPAN 32/34** Siemens offers a line of high performance liquid analyzers in 2- and 4-wire technology, with and without Ex-protection, in field housing or as plug in unit, to measure pH, conductivity, redox potential, and oxygen.

An extensive line of sensors and fittings covers the whole range of applications and measuring ranges. The analyzers are equipped with HART and PROFIBUS DP/PA interfaces and are easy to integrate into automated systems using the Simatic PDM Engineering tool.

Fig. 6 SIPAN Liquid Analyzers

Measuring Locations, Components, Analyzers (1)

MP	Measuring point location	Process medium	Measuring component	Measuring range	Suitable Siemens analyzer
1	Stripper outlet	Waste water	Conductivity	0 ... 250 microS/cm	SIPAN
2	Refiner outlet	Waste water	pH	pH 5 .... pH10	SIPAN
			Conductivity	0 ... 250 microS/cm	SIPAN
3	Primary reactor inlet Fresh/recycle gas mix nozzle	Process gas	O_2 C_2H_4 CH_4 CO_2 C_2H_6 N_2 Ar H_2O C_2H_4O CH_3Cl C_2H_5Cl VinylCl AllylCl	0 ... 10 % 0 ... 40 % 0 ... 100 % 0 ... 10 % 0 ... 1 % 0 ... 20 % 0 ... 10 % 0 ... 2 % 0 ... 3 ppm 0 ... 5 ppm 0 ... 5 ppm 0 ... 5 ppm 0 ... 5 ppm	Quantra Mass Spectrometer
			O_2 + Ar C_2H_4 CH_4 CO_2 C_2H_6 N_2 H_2O EO	0 ... 25 % 0 ... 40 % 0 ... 100 % 0 ... 10 % 0 ... 1 % 0 ... 20 % 0 ... 2 % 0 ... 5 %	MAXUM II Process GC
			C_2H_4 CO_2	0 ... 50 % 0 ... 5 %	ULTRAMAT 6F 2R
			O_2	0 ... 10 %	OXYMAT 6F Ex
			O_2	0 ... 10 %	OXYMAT 6F Ex
			O_2	0 ... 10 %	OXYMAT 6F Ex
			C_2H_4O	0 ... 50 ppm	MAXUM II Process GC
4	Secondary reactor inlet	Process gas	O_2 C_2H_4 CH_4 CO_2 C_2H_6 N_2 Ar H_2O C_2H_4O CH_3Cl C_2H_5Cl VinylCl AllylCl	0 ... 10% 0 ... 40% 0 ... 100% 0 ... 10% 0 ... 1% 0 ... 20% 0 ... 10% 0 ... 2% 0 ... 3 ppm 0 ... 5 ppm 0 ... 5 ppm 0 ... 5 ppm 0 ... 5 ppm	Quantra Mass Spectrometer
			CH_3Cl C_2H_5Cl VinylCl AllylCl	0 ... 5 ppm 0 ... 5 ppm 0 ... 5 ppm 0 ... 5 ppm	MAXUM II Process GC

Table 5 Measuring locations, components and analyzers (1)

Measuring Locations, Components, Analyzers (2)

MP	Measuring point location	Measuring medium	Measuring component	Measuring range	Suitable Siemens analyzer
5	Secondary reactor outlet	Process gas	O_2 C_2H_4 CH_4 CO_2 C_2H_6 N_2 Ar H_2O C_2H_4O CH_3Cl C_2H_5Cl VinylCl AllylCl	0 ... 10 % 0 ... 40 % 0 ... 100 % 0 ... 10 % 0 ... 1 % 0 ... 20 % 0 ... 10 % 0 ... 2 % 0 ... 3 ppm 0 ... 5 ppm 0 ... 5 ppm 0 ... 5 ppm 0 ... 5 ppm	Quantra Mass Spectrometer
			O_2 + Ar C_2H_4 CH_4 CO_2 C_2H_6 N_2 H_2O C_2H_4O	0 ... 25 % 0 ... 40 % 0 ... 100 % 0 ... 10 % 0 ... 1 % 0 ... 20 % 0 ... 2 % 0 ... 5 %	MAXUM II Process GC
			CO_2 CO	0 ... 5 % 0 ... 100 ppm	ULTRAMAT 6F 2R Ex
			O_2	0 ... 10 %	OXYMAT 6F Ex
			O_2	0 ... 10 %	OXYMAT 6F Ex
			O_2	0 ... 10 %	OXYMAT 6F Ex
6	Refiner inlet	Steam	Diss. O_2	0 ... 100 ppm	SIPAN
			Conductivity	0 ... 2 microS/cm	SIPAN
			CH_4	0 ... 300 ppm	FIDAMAT with stripper
7	Desorber inlet	Steam	CH_4	0 ... 300 ppm	FIDAMAT with stripper
8	Converter inlet	Off gas secondary absorber	C_2H_4O	0 ... 50 ppm	MAXUM II Process GC
9	Desorber outlet	Off gas desorber	C_2H_4O	0 ... 50 ppm	MAXUM II Process GC
10	Secondary absorber outlet	Process gas	C_2H_4O	0 ... 50 ppm	MAXUM II Process GC
11	Plant area	Ambient air	UEG	0 ... 25 %	NN
			C_2H_4O	0 ... 1 ppm	Quantra MS
12	Stripper outlet	Waste water	Diss. O_2	0 ... 20 microg/l	SIPAN
13	Refiner outlet	Waste water	Diss. O_2	0 ... 20 microg/l	SIPAN
14	Stripper outlet	Waste water	pH	pH 1 ... pH 14	SIPAN

Table 6 Table 5, continued

Case Study
User Benefits

Process analyzers in an ethylene oxide plant are of extreme significance for correct, safe and efficient plant operation. The analyzers must be of very high quality and reliability and their installation must be performed with a high level of know how and practical engineering expertise.

Siemens Process Analytics

is capable, with respect to both requirements, to offer optimal performance and related benefits to the user, because of

- the respective Siemens analyzers rank on the top of the list of all analyzers available on the market,
- the OXYMAT 6, specifically, provides the outstanding measuring accuracy, reliability, and stability required to operate the process as close as possible to the explosion limit level for highest plant efficiency,
- Siemens Process Analytics has performed successfully numerous deliveries of analyzer systems to ethylene oxide plants including detailed engineering of the analyzer shelters,
- Siemens Process Analytics is part of Siemens Process Automation which provides a strong position for integration of analyzer systems into plantwide automation solutions,

Fig. 7 Analyzer shelter

- Siemens Process Analytics worldwide presence with competence centers, well trained personnel, and technical informations continuously available from the web, for fast and effective customer support wherever it is required, and
- delivery from one hand from first planning up to after sales support over the entire life cycle of the installation provides clear responsibilities and cost transparency.

For further questions please contact our local partner or the following addresses:

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