Process analytics optimizes crude gas processing for the production of fine chemicals at Lonza
Chemical Industry

Fine chemicals are used as active ingredients or additives in the pharmaceutical and food & beverages industries, as well as in agriculture. The cracking of raw materials such as light virgin naphtha or liquefied gas results in intermediate products (ethylene, hydrogen, methane, CO2). At the end of the process chain, these can provide highly pure materials such as niacin (vitamin B3) or pre-stages of vitamins A and E. Process analyzers play an important role in the efficient further processing of cracker products.

Siemens is a leading supplier of process analytics, and has proven its competence for equipping chemical plants with process analytics from planning up to maintenance for many years already. This Case Study describes different applications of process gas chromatographs in a typical fine chemicals production process based on the Lonza acetylene cracker in Visp/Switzerland.

Fine chemicals

Fine chemicals denotes a commercial category for chemical substances. Commonly encountered features include the production in complex syntheses (requiring several reaction steps), in small quantities (only a few tons or kilograms per year) or with a guaranteed degree of purity. This results in a significantly higher price compared to basic chemicals, with typically smaller production quantities. Fine chemicals are required for chemical syntheses and analyses in laboratories, as food additives (e.g. vitamins and minerals) or as raw materials for the production of pharmaceuticals, polymers (additives), detergents and cosmetics.

Lonza is a global player in the field of special chemicals, and produces various fine chemicals products using the high-temperature pyrolysis of light virgin naphtha (LVN) or liquefied petroleum gas (LPG).

In order to monitor the product quality, it is necessary to analyze the chemical composition of the starting materials (LVN, LPG, combustion gas) as well as the crude gas produced in the cracking process. Process gas chromatographs with their high availability have proven their suitability for this purpose. Siemens offers complete system solutions for such applications.
The unique cracker technology: from crude gas generation up to its decomposition

The process plant

Around the year 1915 in Visp/Switzerland (title picture), Lonza began using acetylene as the starting material for the synthesis of important basic chemicals such as acetaldehyde, acetic acid, alcohol (carbide fuel) and further organic products. In the sixties, the preparation of acetylene from calcium carbide was replaced by the high-temperature pyrolysis of light virgin naphtha, which additionally provided hydrogen and ethylene and was the basis for the manufacture of niacin.

Cracker

Today, integrated production processes center around an acetylene/ethylene cracker (Fig. 1). The main raw material is the cracker feed. LPG from nearby refineries is primarily used in addition to LVN.

The naphtha cracking plant can be divided into three functional components:

- Crude gas generation (Montecatini process)
- Crude gas compression
- Gas decomposition (Linde process)

Fig. 2 shows the basic flowchart of the process steps.

Three independent crackers are available for the production of crude gas, of which only two are simultaneously in operation. The LVN or LPG is cracked in the reaction chamber in a temperature range from 2500 to 1200 °C in 2 to 3 thousandths of a second into lowmolecular reaction products (in particular ethylene, propylene, 1,2 and 1,3- butadiene, benzene, toluene, hydrogen, methane and acetylene). The high temperatures are achieved using an oxygen/combustion gas flame at approx. 3,000 °C.

Crude gas scrubbing

The cracked gas is then cooled down to significantly lower temperatures for the subsequent stages by quenching with water. Major contamination such as coke and tar is separated out.

Crude gas compression

The crude gas cooled during the heat recovery is compressed in two steps to 14 bar since the subsequent scrubbing and separating stages are more effective at a higher pressure.

Carbonic acid scrubbing

In an ammonia scrubber, the acidic gases are absorbed at a temperature of approx. 20 °C. In a sequence of individual processes (treatment with activated charcoal, CO₂ purification), the excess technical CO₂ (99.0% purity) is converted into an extremely pure, liquid product with 99.99% CO₂ of food quality. Thus a large portion of the greenhouse gas CO₂ produced in the cracker can be used to advantage.

Crude gas generation

Crude gas compression

Crude gas decomposition

1. LPG analysis with MicroSAM
2. Combustion gas analysis with MAXUM Edition II
3. Crude gas analysis with MAXUM Edition II
4. H₂ analysis with PGC 302

Fig. 2: Basic flowchart of the cracker
Prescrubbing and recovery of acetylene

The acetylene is first scrubbed using a selective solvent. In subsequent rectifying equipment, the acetylene is separated from accompanying substances. Acetylene is required together with ethylene as a raw material for the production of acetaldehyde, from which niacin is produced in subsequent process steps.

Low-temperature gas separation

This is followed by stepwise cooling (-40 °C and -100 °C to -200 °C) of the cracked gas and a number of rectifying steps (Linde process) in which the hydrocarbon mixture is decomposed into fractions with different carbon numbers. The required cooling is generated by an absorption refrigerating plant using ammonia, a methane circuit, and two nitrogen circuits (compressed to 200 bar).

The sequence of separating cuts is of decisive importance for the thermal integration, the equipment requirements, and thus the economy of the process.

Completely different solutions are possible and meaningful here depending on the local conditions, the composition of the cracked gas, and the desired products.

The Lonza cracker with its unique technology thus permits economical C₂ production (acetylene and ethylene) despite its comparatively small size (approx. 50,000 t/yr).

A prerequisite for this is the extensive chemical utilization of the intermediate products produced in the cracker as raw materials for products of higher value within the process chain:

- Niacin (vitamin B3) consumes the major portion of the ethylene production
- Acetylene is used for the production of vitamins A and E as well as scents.
Online analyzers are being used increasingly in the Lonza production plants in Visp. These replace time-consuming and costly laboratory analyses. The process analytics group responsible places particular importance on high availability and minimum maintenance requirements for the analyzers. Siemens process analyzers are in frequent use - for example, various continuous gas analyzers such as 20 ULTRAMAT, 7 OXYMAT, 3 LDS (product descriptions on page 6) and 3 PGC302 (previous model of the MAXUM) are encountered in various sections of the plant.

### The measuring tasks

For further automation of the cracker, the plant owner has therefore decided on an analytical solution comprising two conventional process GCs (MAXUM) and a micro GC (MicroSAM), including the associated sample preparation. The decision in favor of a complementary solution (MAXUM + MicroSAM) was primarily based on the price advantages of the complete solution (Fig. 4). In the decomposition of crude gas, a conventional GC is already being used to analyze the hydrogen purity. Fig. 5 lists the measuring tasks of the process GCs at the various measuring points within the plant.

### Quality analysis of the cracker feed

MicroSAM provides information on the product composition and thus on the quality of the LPG fed to the plant. In the case of deviations from normal operation, the plant owner can rapidly investigate the possible causes.

### Analysis of combustion gas

The oxygen requirements of the combustion gas are calculated using the analytical data provided by the MAXUM. The device is used as a backup for the continuous oxygen requirement measurement.

### Analysis following crude gas scrubbing

The MAXUM provides information on the composition of the crude gas during scrubbing. The cracker is set according to the C₂ ratio. Parallel to an IR photometer, the process GC provides important supplementary information, particularly when deviations occur in the process.

### Analysis of hydrogen purity

During the decomposition of crude gas, one of the products is hydrogen whose impurities have to be measured using process GC.

### The solution

#### MAXUM Edition II

The MAXUM is the technological leader in process gas chromatography. Its technical properties provide it with exceptional versatility. Every analytical task required can be solved by the MAXUM with the best analytical results and lowest possible costs at the same time:

- Compression using a modular concept saves investment costs - 2 to 3 GCs in one MAXUM
- Parallel chromatography
- For faster analyses and increased reliability
- Airless oven for reduced consumption of instrument air and energy
- Many types of detector (TCD, FID, FPD, HID, ECD, PID) for flexible analytical solutions
- 3 types of detector possible in one MAXUM, multi-channel TCD: 2, 4 or 8 channels
- Valveless column switching for reduced maintenance requirements - no moving parts in the sample path
- EPCs and Ethernet already included in the standard package (no extra costs)

The applications with MAXUM as described here apply the above-mentioned concepts on the basis of micropacked column technology, valve switching and multi-channel TCD detectors.
Increased customer benefits through a complementary solution with MAXUM and MicroSAM

MicroSAM

State-of-the-art silicon-based micromechanical components allow miniaturization and increased performance at the same time. As a result of its small dimensions (the size of a football) and rugged design (IP65, NEMA4X, explosion protection through flameproof enclosure), the extremely compact device can be installed at any position in a plant, for example directly at the sampling point of a reactor, and offers decisive advantages:

- Analyzer in the analysis container for space-saving installation and low investment costs
- Installation of analyzer close to sampling point for fast analyses and low investment costs - no air-conditioned analysis container required
- Replacement of standard parts instead of repairs saves money - no stocking of spare parts/servicing
- Remote maintenance results in lower maintenance costs - the analyzer does not have to be inspected on site
- Extremely low consumption of auxiliary media results in reduced operating costs (current consumption and instrument air)

During the quality analysis of the cracker feed, the original liquid sample is continuously vaporized by the sample conditioning system, and injected as a gas into the GC. The sample composition is determined by an analytical module with narrow-bore capillary columns connected in series and having different separating characteristics and in-line micro TCDs.

### Proof of performance

#### Calibration trend demonstrates high long-term stability

Process GCs must be regularly calibrated directly with external calibration media or indirectly using a parallel laboratory analysis. The calibration frequency largely depends on factors such as the sample composition or ambient conditions, or the philosophy of the plant owner. Calibration cycles usually vary between several days and several weeks. In the applications presented here, the calibration was applied for validation purposes in order to check the repeatability of the measurements over a longer period and thus the reliable operation of a process GC.

Fig. 6 uses the MAXUM for crude gas analysis as an example of the long-term stability of the GC. The permissible tolerances averaged over a period of 44 months are a factor of 10 below the permissible values.

The deviations for the MicroSAM are also within the permissible tolerances (Fig. 7). As a result of this high long-term stability, the calibration cycles for both chromatographs can be extended to two months without loss of reliability.

### User benefits

- Proven and reliable standard technology with high long-term stability
- High reliability through comprehensive self-diagnostics
- High cost efficiency, both
  - in the operating costs through ...high device availabilities > 99.5%), extended calibration periods (every 2 months), low costs for consumable media (supply gases, air, power) and reduced servicing requirements
  - and in the investment costs through ...complementary solution with MAXUM and MicroSAM (small space requirement of MicroSAM allows integration of additional analyzers in the analysis container)

In addition, costs are saved during process operation.

### Table: Measured components

<table>
<thead>
<tr>
<th>Measured components</th>
<th>Concentration in the calibration gas [in % abs.]</th>
<th>Permissible tolerance [± in %]</th>
<th>Maximum actual deviation [± in %]</th>
<th>Average actual deviation [± in %]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>31</td>
<td>2</td>
<td>0.9</td>
<td>0.27</td>
</tr>
<tr>
<td>Methane</td>
<td>13</td>
<td>1</td>
<td>0.7</td>
<td>0.1</td>
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<tr>
<td>CO</td>
<td>18</td>
<td>1.25</td>
<td>0.75</td>
<td>0.14</td>
</tr>
<tr>
<td>CO₂</td>
<td>13</td>
<td>1.25</td>
<td>0.42</td>
<td>0.08</td>
</tr>
<tr>
<td>Ethane</td>
<td>1</td>
<td>0.15</td>
<td>0.9</td>
<td>0.27</td>
</tr>
<tr>
<td>Ethene</td>
<td>14</td>
<td>1</td>
<td>0.7</td>
<td>0.1</td>
</tr>
<tr>
<td>Ethine</td>
<td>8</td>
<td>0.5</td>
<td>0.75</td>
<td>0.14</td>
</tr>
<tr>
<td>Propene</td>
<td>2</td>
<td>0.15</td>
<td>0.42</td>
<td>0.08</td>
</tr>
</tbody>
</table>

Fig. 6: Checking of long-term stability of the calibration analyses using example of MAXUM for crude gas analysis. Period 27.01.2003 - 08.09.2006 (3 years 8 months)

Fig. 7: Analysis container with process analyzers
Siemens Process Analytics is a leading provider of process analyzers and process analysis systems. We offer our global customers the best solutions for their applications based on innovative analysis technologies, customized system engineering, sound knowledge of customer applications and professional support. And with Totally Integrated Automation (TIA). Siemens Process Analytics is your qualified partner for efficient solutions that integrate process analysers into automations systems in the process industry.

From demanding analysis tasks in the chemical, oil and gas and petrochemical industry to combustion control in power plants to emission monitoring at waste incineration plants, the highly accurate and reliable Siemens gas chromatographs and continuous analysers will always do the job.

Siemens Process Analytics offers a wide and innovative portfolio designed to meet all user requirements for comprehensive products and solutions.

Our Products

The product line of Siemens Process Analytics comprises

- extractive and in-situ continuous gas analyzers (fig. 8-11)
- process gas chromatographs (fig. 12-13)
- sampling systems
- auxiliary equipment

Analyzers and chromatographs are available in different versions for rack or field mounting, explosion protection, corrosion resistant etc.

A flexible networking concept allows interfacing to DCS and maintenance stations via 4-20 mA, PROFIBUS, OPC, Modbus or industrial ethernet.
Product Scope
Siemens Continuous Gas Analyzers and Process Gas Chromatographs

Extractive Continuous Gas Analyzers (CGA)

**ULTRAMAT 23**
The ULTRAMAT 23 is a cost-effective multicomponent analyzer for the measurement of up to 3 infrared sensitive gases (NDIR principle) plus oxygen (electrochemical cell). The ULTRAMAT 23 is suitable for a wide range of standard applications. Calibration using ambient air eliminates the need of expensive calibration gases.

**CALOMAT 6/62**
The CALOMAT 6 uses the thermal conductivity detection (TCD) method to measure the concentration of certain process gases, preferably hydrogen. The CALOMAT 62 applies the TCD method as well and is specially designed for use in applications with corrosive gases such as chlorine.

**OXYMAT 6/61/64**
The OXYMAT 6 uses the paramagnetic measuring method and can be used in applications for process control, emission monitoring and quality assurance. Due to its ultrafast response, the OXYMAT 6 is perfect for monitoring safety-relevant plants. The corrosion-proof design allows analysis in the presence of highly corrosive gases. The OXYMAT 61 is a low-cost oxygen analyser for standard applications. The OXYMAT 64 is a gas analyzer based on ZrO₂ technology to measure smallest oxygen concentrations in pure gas applications.

**FIDAMAT 6**
The FIDAMAT 6 measures the total hydrocarbon content in air or even in high boiling gas mixtures. It covers nearly all requirements, from trace hydrocarbon detection in pure gases to measurement of high hydrocarbon concentrations, even in the presence of corrosive gases.

**ULTRAMAT 6**
The ULTRAMAT 6 uses the NDIR measuring principle and can be used in all applications from emission monitoring to process control even in the presence of highly corrosive gases. ULTRAMAT 6 is able to measure up to 4 infrared sensitive components in a single unit.

ULTRAMAT 6 / OXYMAT 6
Both analyzer benches can be combined in one housing to form a multi-component device for measuring up to two IR components and oxygen.

Process Gas Chromatographs (Process GC)

**MAXUM edition II**
MAXUM edition II is very well suited to be used in rough industrial environments and performs a wide range of duties in the chemical and petrochemical industries and refineries. MAXUM II features e.g. a flexible, energy saving single or dual oven concept, valveless sampling and column switching, and parallel chromatography using multiple single trains as well as a wide range of detectors such as TCD, FID, FPD, PDHID, PDECD and PDPID.

**MicroSAM**
MicroSAM is a very compact explosion proof micro process chromatograph. Using silicon-based micromechanical components it combines miniaturization with increased performance at the same time. MicroSAM is easy to use and its rugged and small design allows mounting right at the sampling point. MicroSAM features drastically reduced cycle times, provides valveless sample injection and column switching and saves installation, maintenance, and service costs.

**SITRANS CV**
SITRANS CV is a micro process gas chromatograph especially designed for reliable, exact and fast analysis of natural gas. The rugged and compact design makes SITRANS CV suitable for extreme areas of use, e.g. off-shore exploration or direct mounting on a pipeline. The special software "CV Control" meets the requirements of the natural gas market, e.g. custody transfer.

**In-situ Continuous Gas Analyzers (CGA)**

**LDS 6**
LDS 6 is a high-performance in-situ process gas analyzer. The measurement (through the sensor) occurs directly in the process stream, no extractive sample line is required. The central unit is separated from the sensor by using fiber optics. Measurements are carried out in realtime. This enables a pro-active control of dynamic processes and allows fast, cost-saving corrections.
Analytical solutions are always driven by the customer’s requirements. 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 (fig. 15). This includes also signal processing and communications to the control room and process control system.

We rely on many years of world-wide experience in process automation and engineering and a collection of specialized knowledge in key industries and industrial sectors. We provide Siemens quality from a single source with a function warranty for the entire system.

Read more in chapter “Our services”.

Siemens Process Analytics provides networking solutions to meet the demands of both objectives.
Siemens Process Analytics – Our Services

Siemens Process Analytics is your competent and reliable partner worldwide for Service, Support and Consulting.

Our resources for that are

• **Expertise**
  As a manufacturer of a broad variety of analyzers, we are very much experienced in engineering and manufacturing of analytical systems and analyzer houses. We are familiar with 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 our strategically located centers of competence in Germany, USA, Singapore, Dubai and Shanghai, we are globally present and acquainted with all respective local and regional requirements, codes and standards. All centers are networked together.

**Service portfolio**

Our wide portfolio of services is segmented into Consulting, Support and Service. It comprises really all measures, actions and advises that may be required by our clients throughout the entire lifecycle of their plant:

- Site survey
- Installation check
- Functionality tests
- Site acceptance test
- Instruction of plant personnel on site
- Preventive maintenance
- On site repair
- Remote fault clearance
- Spare part stock evaluation
- Spare part management
- Professional training center
- Process optimisation
- Internet-based hotline
- FEED for Process Analytics
- Technical consulting

Fig. 18 Portfolio of services provided by Siemens Process Analytics

**FEED for Process Analytics**

Front End Engineering and Design (FEED for PA) is part of the planning and engineering phase of a plant construction or modification project and is done after conceptual business planning and prior to detail design. During the FEED phase, best opportunities exist for costs and time savings for the project, as during this phase most of the entire costs are defined and changes have least impact to the project. Siemens Process Analytics holds a unique blend of expertise in analytical technologies, applications and in providing complete analytical solutions to many industries.

Based on its expertise in analytical technology, application and engineering, Siemens Process Analytics offer a wide scope of FEED services focused on analyzing principles, sampling technologies, application solutions as well as communication system and given standards (all related to analytics) to support our clients in maximizing performance and efficiency of their projects.

Whether you are plant operators or belong to an EPC Contractor you will benefit in various ways from **FEED for Process Analytics** by Siemens:

- Analytics and industry know how available, right from the beginning of the project
- Superior analyzer system performance with high availability
- Established studies, that lead to realistic investment decisions
- Fast and clear design of the analyzer system specifications, drawings and documentation
- Little project management and coordination effort, due to one responsible contact person and less time involvement
- Additional expertise on demand, without having the costs, the effort and the risks of building up the capacities
- Lowest possible Total Costs of Ownership (TCO) along the lifecycle regarding investment costs, consumptions, utilities supply and maintenance
Notes:
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