Use of Process Analytics in Phthalic Anhydride-production plants.

Siemens provides the suitable analyzers and extensive application know how

Phthalic Anhydride

Phthalic anhydride, in short PA PA, $C_6H_4(CO)_2O$ is an anhydride of the Phthalic acid. This organic compound is an important starting material to produce plasticisers (e.g. PVC) and synthetic resins, but also dyes and pigments. PA has been produced first in 1872 by oxidation of naphtalene. Ithas been used commercially since that time and is comparable in its importance to hydrochloric acid. The World production today is several million tons per year and depends strongly on the demand for PVC. Raw material for PA was first only coal-tar naphtalene (which has been oxidized by air) and thus depended on the production of coke, which could not follow the increasing demand for naphtalene. Since 1960 a shift took place therefore in raw material base from naphthalene to o-Xylene, which is available in adequate quantities from cracking.

Production process of PA has been improved several times, a breakthrough that led to high quality production was the development of the gas-phase oxidation of naphtalene or o-xylene in an air stream using different types of catalysts.

Production of Phthalic Anhydride

As already mentioned, o-Xylene is used as raw material for the production of PA since the 1960s and is available in adequate quantities from cracking plants and refineries. Nowadays new PA plants can process naphtalene and o-Xylene or mixtures of the two.
Production processes and plants differ in details depending on the manufacturer and plant engineering. The oxidation of o-Xylene is performed at a temperature of 360 – 390 °C and using a catalyst (most common is V2O5) according to $C_6H_4(CH_3)_2 + 3O_2 = C_8H_4O_3 + 3H_2O$.

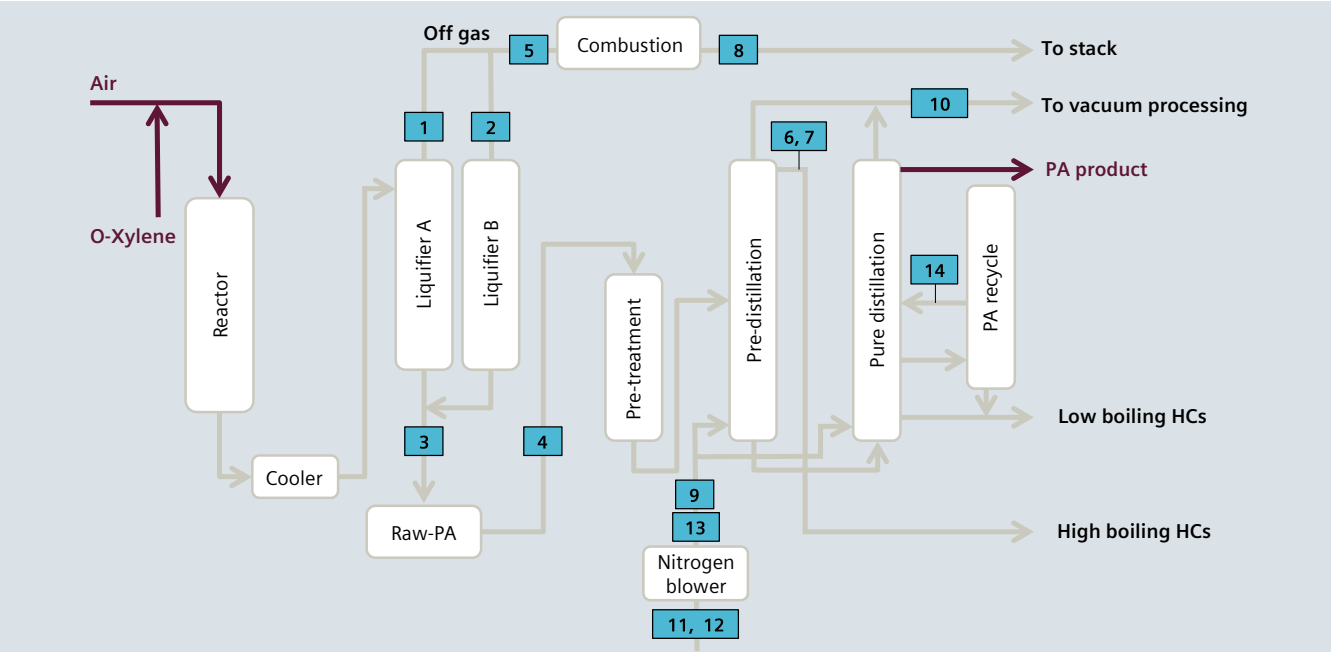

Fig. 1: PA production flow diagram (exemplary)

The process steps (fig. 1 and table 1)

- Introduction of preheated o-Xylene into a compressed stream of hot air.
- Flow of the o-Xylene/air mixture through a tubular reactor with exothermic oxidation on a selective catalyst. A molten salt is used to remove generated heat and fed to a steam generator, where high pressure steam is produced and utilized in the plant.
- Pre-cooling of the gases leaving the reactor and feeding to a condensor system.
- Condensation of PA as a solid. The condensers are cooled and heated in a switching cycle.
- Melting of the solid PA during the heating cycle of the switch condenser and transfer as crude material into a storage tank.
- Post treatment and purification by two-stage distillation.
- Incineration of the waste gas.

Process Analytics in PA plants

Process analyzers are a part of field instrumentation in modern PA production plants. They perform different process application tasks which are supported by Siemens Process Analytics with its high performance MAXUM edition II process gas chromatograph as well as the OXYMAT and ULTRAMAT continuous gas analyzers.

One important objective of PA technology is to reduce energy consumption as far as possible. This can be achieved by increasing the o-Xylene concentration in the process air, which, however, may cause, that the limit of flammability of the o-Xylene/air mixture is exceeded with the danger of generating explosive conditions.

Therefore, the entire production process must be controlled by analyzing oxygen and some other gas components continuously with highest care and, at several measuring locations, with high redundancy (application “Plant safety”). Other applications refer to product composition (Product quality) and compliance with emission limits (Emission control).

Instrumentation

Process Gas Chromatography

MAXUM edition II
Process Gas Chromatograph

MAXUM edition II represents the top technology in process gas chromatography for analyzing liquids and vapor process samples. Unparalleled product features deliver high versatility and the best possible analytical results at low operating costs through

- Broad selection of components such as injectors, ovens, detectors or tools for column switching
- Optimal dosage of liquid samples by using a special liquid dosing valve
- Reliable trace analysis through very sensitive detectors
- High cost efficiency by combining single and double ovens and using the modular oven concept
- Continuous real time monitoring of networked analyzers by a workstation (fig. 3)

Sampling point	Medium and Measuring position	Measuring task / Application	Meas. comp.	Meas. Range	Siemens Analyzer
1	**Process gas** Liquifier A Outlets	Plant safety	O_2	0 – 10 %	OXYMAT (3 times red.)
			CO	0 – 2 %	ULTRAMAT
			CO_2	0 – 10 %	
		Product quality	Var. HC	ppm	MAXUM edition II
2	**Process gas** Liquifier B Outlets	Plant safety	O_2	0 – 10 %	OXYMAT (3 times red.)
			CO	0 – 2 %	ULTRAMAT
			CO_2	0 – 10 %	
		Product quality	Var. HC	ppm	MAXUM edition II
3	**Raw PA** Liquifier A+B Outlets	Plant safety	O_2	0 – 10 %	OXYMAT (3 times red.)
			CO	0 – 2 %	ULTRAMAT
			CO_2	0 – 10 %	
		Product quality	Var. HC	ppm	MAXUM edition II
4	**Raw PA Outlet**	Product quality	Var. HC	ppm	MAXUM edition II
5	Off gas from liquifiers	Plant safety	O_2	0 – 8 %	OXYMAT
6	Process medium outlet		O_2	0 – 8 %	OXYMAT
7	Process medium outlet		O_2	0 – 8 %	OXYMAT
8	Off gas downstream of wasteincinerator	Plant safety Emission control	O_2	0 – 15 %	OXYMAT
9	Blower at suction location	Plant safety	O_2	0 – 8 %	OXYMAT
10	Seperator		O_2	0 – 8 %	OXYMAT
11	Nitrogen compressor intake		O_2	0 – 8 %	OXYMAT
12	Service connection Nitrogen compressor		O_2	0 – 8 %	OXYMAT
13	Service connection Nitrogen compressor Outlet		O_2	0 – 8 %	OXYMAT
14	Process stream to recycling	Plant safety	O_2	0 – 8 %	OXYMAT

Tab. 1: Sampling Points, measuring tasks and analyzers

Kontinuierliche Gasanalytik

Paramagnetic oxygen gas analyzer OXYMAT

The function of the OXYMAT analysis module is based on the paramagnetic alternating pressure method and is used to measure oxygen in gases.

- High reliability and very short response time due to simple and robust design without moving parts
- Small measuring ranges (0 to 0.5 % oder 99.5 bis 100 % O_2)
- Physically suppressed zero point, e. g. 98 or 99.5 to 100 % O_2
- correction of pressure variations in sample gas using an internal pressure sensor
- applicable to highly corrosive gases due to the use of highly resistant sample chamber material
- SIL 2 certification enabling safety oriented applications in protection systems

Fig. 2: MAXUM edition II

Fig. 3: Gas Chromatograph Portal Workstation Software

Fig. 4: OXYMAT and ULTRAMAT in different versions.

NDIR gas analyzer ULTRAMAT

The ULTRAMAT analysis module uses the NDIR dual-beam method. It selectively measures gases with an absorption band in the infrared wavelength range.

- High measuring precision, very low signal noise and no microphony effects by using a sensor without moving parts (micro-flow sensor)
- High selectivity due to two-layer detector: high measuring precision in complex gas mixtures
- High operational reliability and life time thanks to an extremely robust mechanical design
- High measuring precision also in case of complex sample gas mixtures

Two analyzers modules can be fitted in the available housing types, which offer many advantages for continuous gas analytics: combined measurements and interfering gas corrections in one device, fast and easy replacing of modules, and thus particularly economic retrofitting or conversion, and operating of all modules effortlessly by just one display unit.

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