# Utilizing online chemical analysis to optimize propylene oxide production


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Propylene oxide (PO) is a major industrial product with a yearly global production of more than 7 million tons. PO is used in assorted industrial applications, though mainly for the production of polyols, the building blocks for polyurethane plastics. Several production methods exist, with and without co-products. This white paper lays out opportunities to optimize PO production for safer and more efficient processes, higher quality products, and substantial time savings by using online process analysis instead of laboratory measurements.

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# Propylene oxide

## Introduction

Propylene oxide (PO) is a colorless and extremely flammable liquid known by several names—epoxypropane, methyloxirane, and epihydrin, to list a few.[1,2]


PO is derived from crude oil and is used in several industrial applications, however it is mainly consumed for the production of polyols which are the building blocks for polyurethane plastics. The primary reactant used is propene (propylene), though there are several different production processes currently in use on the market.[1–6]

## Derivative markets

Many different markets utilize PO to manufacture other products. Some of the top derivative markets are:

- polyols
- propylene glycol
- propylene glycol ethers

## Various uses of propylene oxide

PO is a major industrial product with a global production of more than 7 million tons per year.[4] Approximately 70% is used to make polyether polyols, which are raw materials used in production of polyurethane.[1,3–6] Another 20% is utilized to make propylene glycol, used as an additive in cosmetics and refined foods,[4–6] as well as in the production of unsaturated polyesters.[1,6] The final 10% or so is used to create propylene glycol ether solvents and other products.[2,6]

## Production methods

There are several production processes available, however the majority of PO is still co-produced along with styrene monomer (approximately one third of PO production worldwide).[1] Other methods include the chlorohydrin process, epoxidation of propylene with hydrogen peroxide, epoxidation of propylene with organic peroxides, and even epoxidation using molten salts.[1–6]

## Process depends on market needs

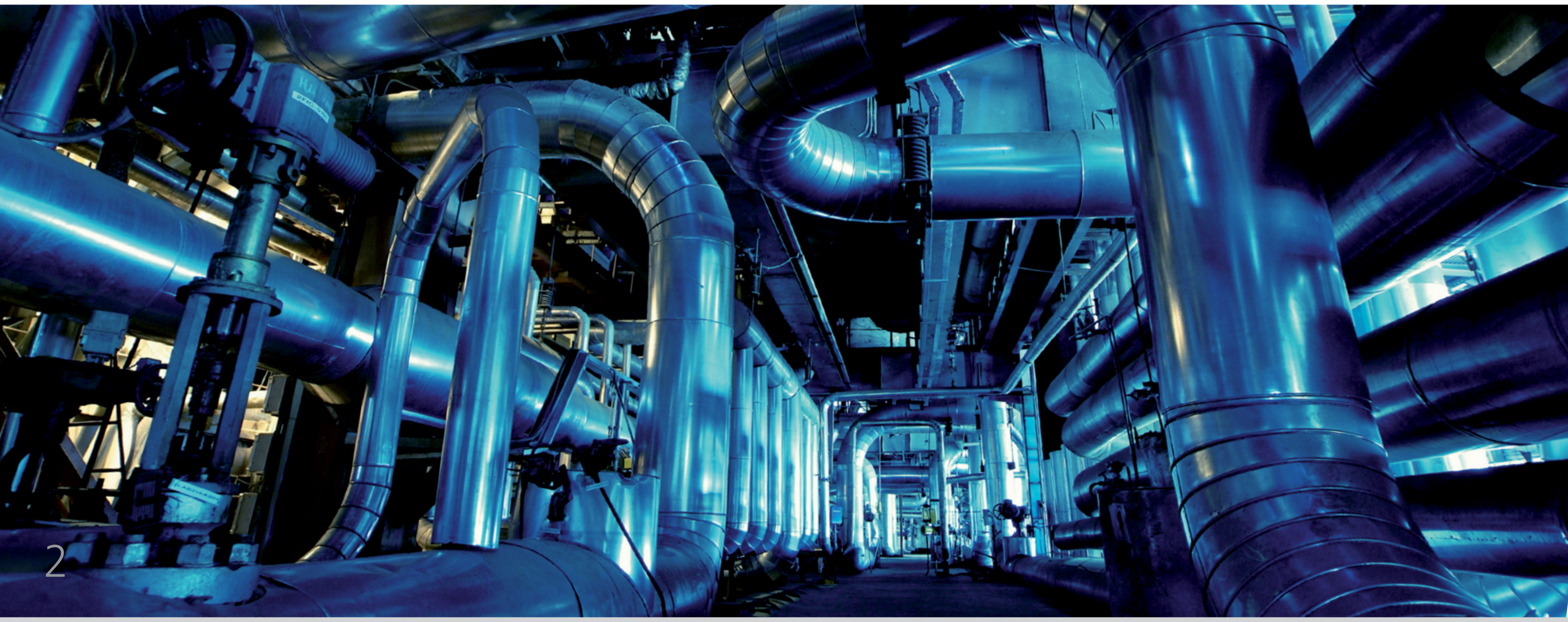
PO production methods are available both with and without byproduct materials. Depending on the market for these byproducts, one or more processes may be in major use globally at any one time.

**Processes with co-products:**

- Chlorohydrin (CH-PO)
- Styrene (SM-PO)
- Methyl *tert*-butyl ether / *Tert*-butyl alcohol (MTBE-PO / TBA-PO)

**Derivative-free processes:**

- Cumene (CU-PO)
- Hydrogen peroxide (HP-PO)



## Overview of the major PO production processes

# The chlorohydrin process (chlorine process)

The first large-scale process developed for the production of propylene oxide was based on the chlorohydrin route in the earlier part of the 20th century. The chlorohydrin process has been in decline since the 1950's, though it still accounts for a significant proportion of PO production volume globally.[3,4] High costs are involved both on the environment from the massive amounts of wastewater and for utilities to generate the large amounts of electricity needed to produce the feedstock.

### Production method

This method of PO manufacture is shown in **Figure 1** and **Figure 2**. The selectivity for producing propylene oxide is near 90%, however hundreds of kilograms of byproducts without any significant sales market are obtained per ton of PO.[1–3]

### Waste from CH-PO process

Wastewater resulting from the chlorohydrin process needs complicated and elaborate treatment methods. Salt resulting from the dechlorination procedure can be disposed of in the wastewater as well, though it is possible through chlor-alkali electrolysis to recover caustic and chlorine (at increased cost).[3]

### CH-PO – Byproducts produced per ton of PO:[1–5]

- 100–150 kg 1,2-dichloropropane
- 2.1 tons NaCl or 2.2 tons $CaCl_2$
- 40 tons wastewater

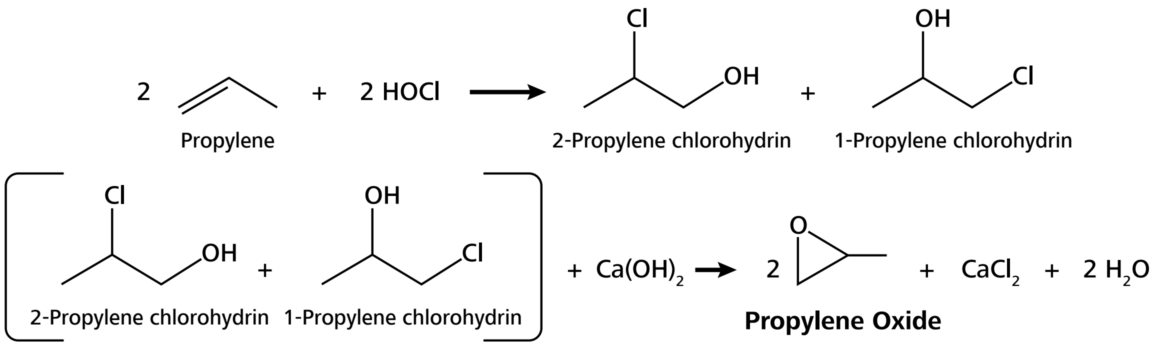



**Figure 1.** Chemical process behind the chlorohydrin route to produce propylene oxide. Adapted from Bernhard et al.[3]

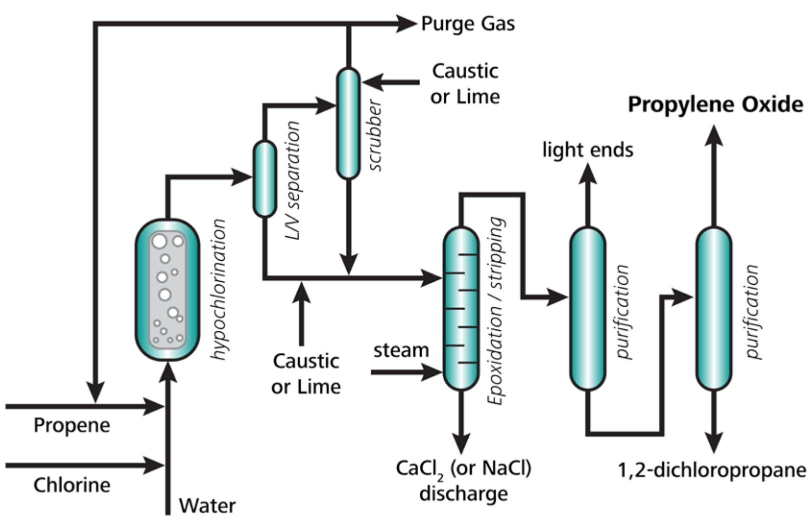



**Figure 2.** Schematic process diagram outlining the chlorohydrin method for co-production of propylene oxide. Adapted from Nijhuis et al.[2]

## The styrene process (organic peroxide process)

### Hydroperoxide process for PO production

The hydroperoxide process involves oxidation of propylene to PO via use of an organic hydroperoxide, with a resulting alcohol as a co-product. Commercially, there are two different hydroperoxides in use: ethylbenzene hydroperoxide (**SM-PO process**) and *tert*-butyl hydroperoxide (**TBA/MTBE-PO process**).

### Manufacturing styrene monomer

Styrene monomer is one of the most important large-volume commodity chemicals, with more than 27 million tons produced worldwide each year.[4] Two major production routes are in use—the ethylbenzene (dehydrogenation) process, which is the most common, and via co-production with propylene oxide (SM-PO process).[1–5] The SM-PO process along with the TBA/MTBE-PO process account for **more than 40%** of the global total capacity of PO.[3]

### Production method: Styrene Monomer and PO

The SM-PO process, which is outlined in **Figure 3** and **Figure 4**, creates styrene, with propylene oxide as the major co-product.[2–5] Recovered feedstocks are recycled back into the process. This method produces 2–2.5 tons of styrene monomer per ton of PO.[3–5]

### Principal sources of emissions to water:[4]

- acid purge from the oxidation unit
- aqueous stream from epoxidation caustic wash
- aqueous stream from styrene monomer production and purification

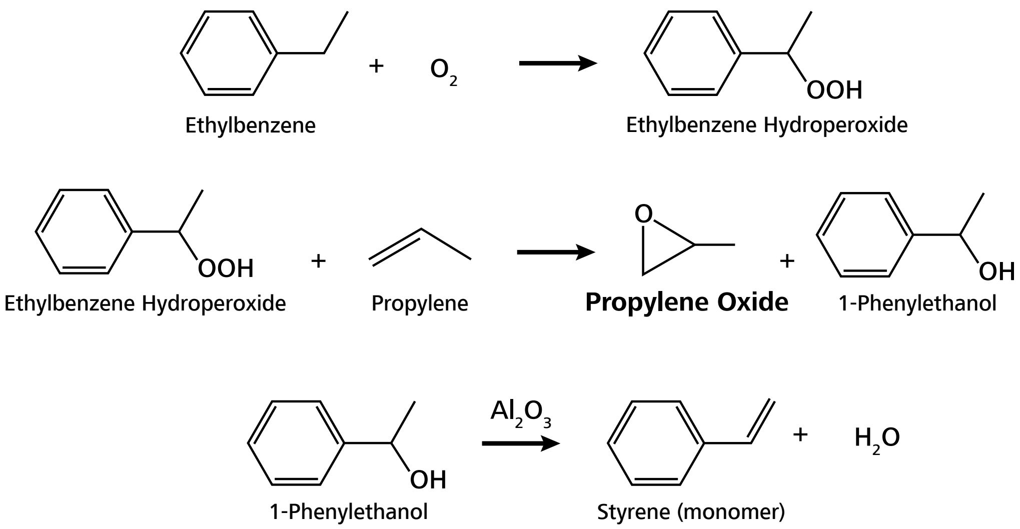



**Figure 3.** Chemical process behind the styrene (SM-PO) route to produce propylene oxide.[3,4]

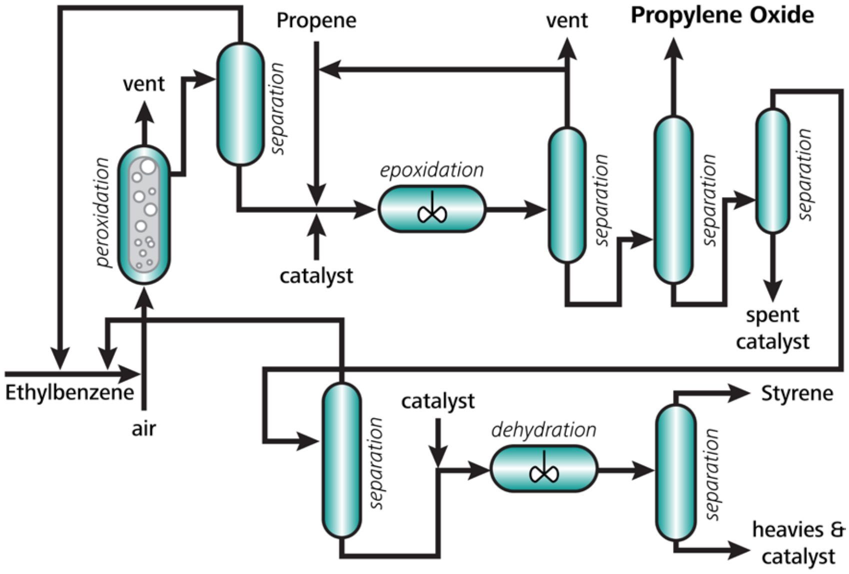



**Figure 4.** Schematic process diagram outlining the styrene method for co-production of propylene oxide. Adapted from Nijhuis et al.[2]

## SM-PO process conditions

- Primary reactor conditions: 120–150 °C, 2–3 bar[1–4]
- Intermediate (EBHP) reaction conditions with propene over catalyst: between 90–130 °C, 15–60 bar[1–4]
- Total selectivity with respect to PO: 90% or more[1–3]
- 2.2–2.5 tons styrene produced per ton of PO[3,5]
- Process water condensate is stripped, organics recycled back in to process, purified water as boiler feed
- Crude epoxidate stream is washed with caustic then distilled to recover more PO from the process

**Related application note:**
Monitoring of 4-tert-butylcatechol in styrene in accordance with ASTM D4590: AN-PAN-1027
https://www.metrohm.com/en/applications/AN-PAN-1027

## The TBA / MTBE process (organic peroxide process)

### Co-production with TBA

The second commercial hydroperoxide process in use today is the propylene oxide–*tert*-butyl alcohol (PO-TBA) process, also known as TBA-PO or MTBE-PO / PO-MTBE. **Figure 5** and **Figure 6** summarize the production process.

### Uses of *tert*-butanol and methyl *tert*-butyl ether

*Tert*-butanol (TBA) is used as a solvent and as a denaturant in ethanol. It is also used as an intermediate in the production of methyl *tert*-butyl ether (MTBE), which is the reasoning behind the many names for this process. The TBA co-product is converted directly to MTBE with methanol in the presence of a catalyst.

The majority of MTBE is utilized by refineries to increase the octane number in gasoline as an additive (oxygenate) to extend the lifespan of engines, especially in heavy-duty machinery.[1–5] Further processing of MTBE leads to the production of isobutylene,[1,5] which is used to make polyisobutylene (synthetic rubber) and the acrylic resin methyl methacrylate (MMA).

Regulations around the contamination of groundwater threaten the estimated market growth for MTBE.

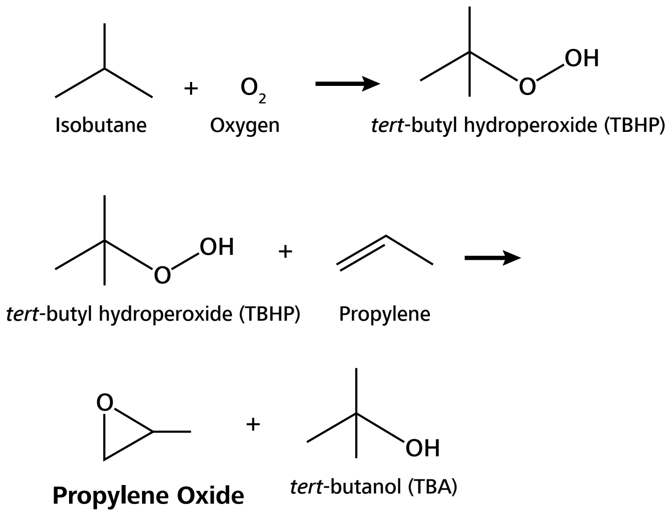



**Figure 5.** Chemical process behind the PO-TBA (also known as TBA-PO or MTBE-PO / PO-MTBE) route to produce propylene oxide.[1]

### TBA / MTBE (TBA-PO / MTBE-PO) process conditions

- Primary reactor conditions: 95–150 °C, 25–55 bar[1–3]
- Intermediate (TBHP) reaction conditions with propene over catalyst: between 90–130 °C, 15–60 bar[1–3]
- Total selectivity with respect to PO: 90–95%[1,3]
- 2.5–3.5 tons *tert*-butanol produced per ton of PO[3]
- Process water condensate is stripped, organics recycled back in to process, purified water as boiler feed

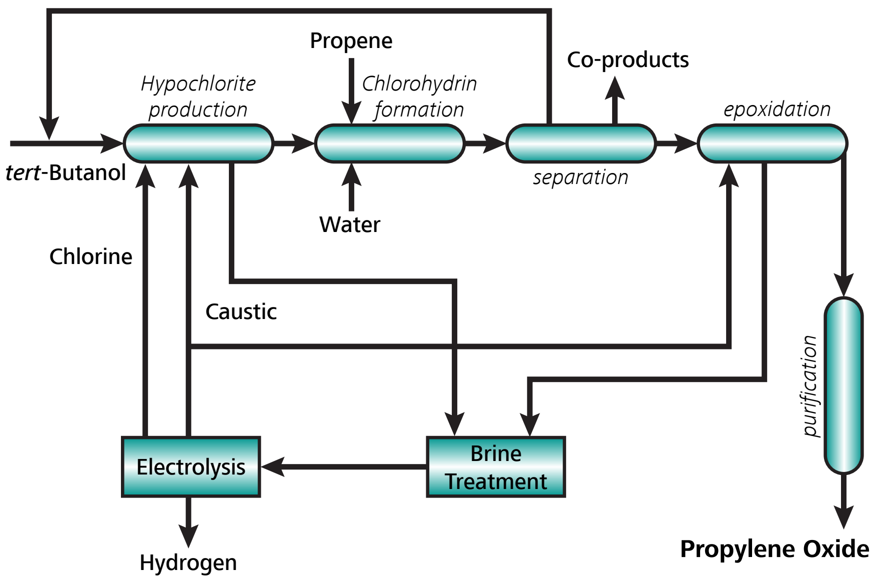



**Figure 6.** Schematic process diagram outlining the propylene oxide-*tert*-butyl alcohol (PO-TBA, also TBA-PO, MTBE-PO, or PO-MTBE in literature) co-production method. Adapted from Trent.[1]

## Byproduct-free methods to produce PO

The following processes utilize (organic) peroxides for the epoxidation of propylene to produce PO with **only water** as a byproduct.

# The cumene process (organic peroxide process)

### The cumene process (organic peroxide process)

The cumene to propylene oxide process (CU-PO) was developed by Sumitomo Chemical. This PO production method is shown in **Figure 7** and **Figure 8**.

### Advantageous production method

Besides not being dependent on a separate co-product market, yet another advantage for CU-PO over SM-PO (and other methods of PO production with byproducts) is the stability of the hydroperoxide intermediate (CMHP).[5]

### Cumene in the chemical industry

Cumene is an aromatic hydrocarbon which is mainly used (in its purified form) to produce cumene hydroperoxide (CMHP) —a valuable intermediate for several other industrially processed chemicals. Among these are phenol and acetone, detailed more in the related process application note, and later on in the applications section of this paper.

**Related application note:**
Determination of sulfuric acid in acetone and phenol: AN-PAN-1008
https://www.metrohm.com/en/applications/AN-PAN-1008

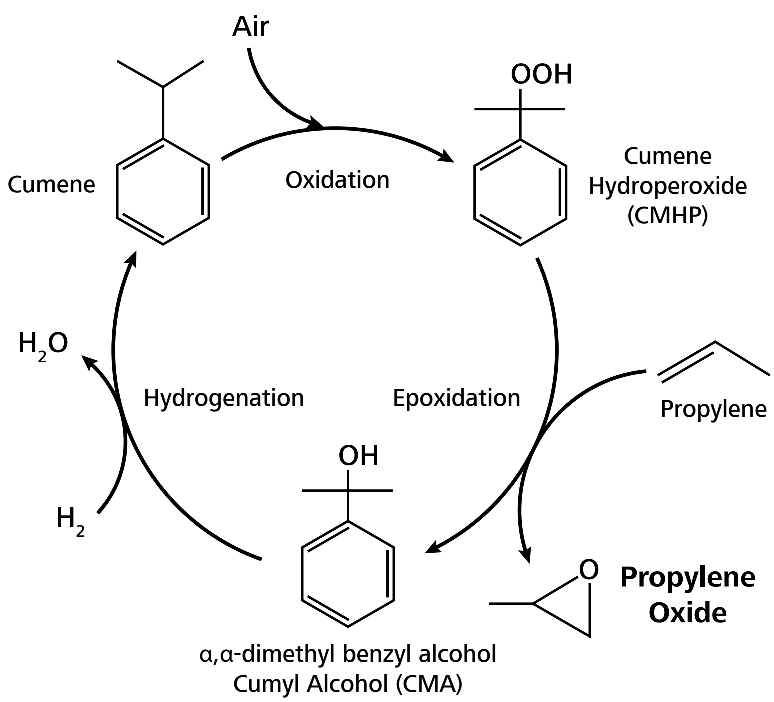



**Figure 7.** Chemical process behind the cumene route to produce propylene oxide.[5,7]

### Cumene (CU-PO) process conditions

- Oxidation conditions: atmospheric pressure (or up to 7 bar with autocatalysis)[4], atmospheric $O_2$, 80–120 °C[3–5]
- Cumene is purified and recycled within the process[2,4,5]
- Unreacted propene is also recovered and recycled[5]
- Temperature must be regulated (heat exchangers) to avoid secondary reactions & runaway epoxidation
- Total selectivity with respect to PO: over 90%[2]

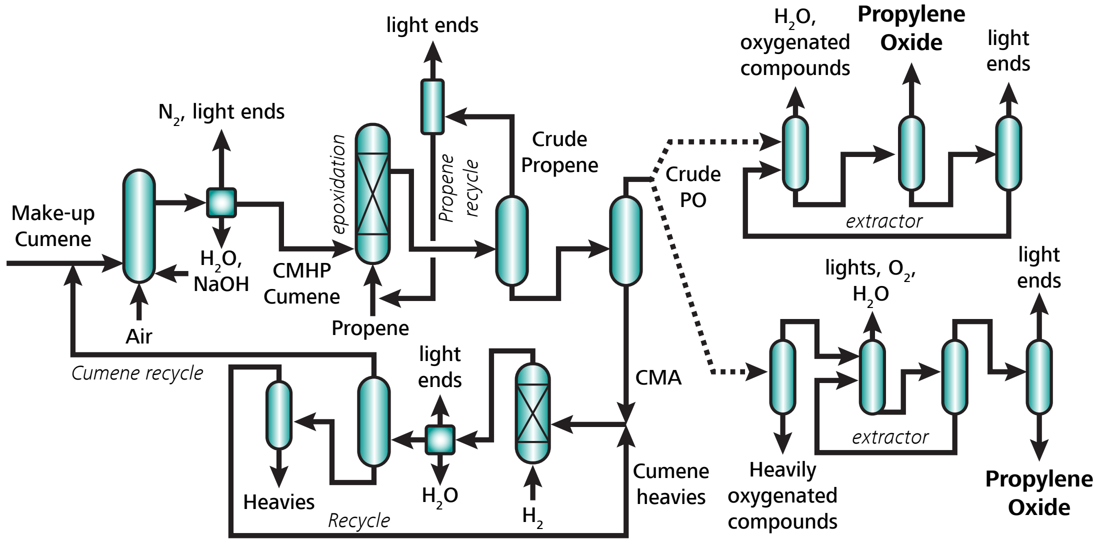



**Figure 8.** Schematic process diagram outlining the cumene-propylene oxide (CU-PO) method for byproduct-free production of PO. Adapted from Nemeth and Bare.[7]

# The hydrogen peroxide process

### Harnessing the oxidation potential of $H_2O_2$

It was not until the 1990's that hydrogen peroxide ($H_2O_2$) could be used directly as an oxidizing agent in the production of propylene oxide (now known as the HP-PO process).[3] Conventional catalysts were found to be insufficient, and thus a new catalyst (titanium silicalite-1, TS-1, **Figure 9**) was synthesized to limit and suppress any secondary reactions.[1–3]

### Epoxidation of propene with hydrogen peroxide

The details of the HP-PO process are shown in **Figure 10** and **Figure 11**. Unreacted propene is recycled to the main reactor after removal of any traces of $O_2$ for safety reasons.

The HP-PO process is nearly quantitative in producing PO from a complete conversion of $H_2O_2$. The selectivity for PO in this process has been measured at **more than 98%**.[2,3] The effluent from this process must be monitored for traces of substances such as methoxypropanols and glycols prior to being discharged to a wastewater treatment plant (WWTP).

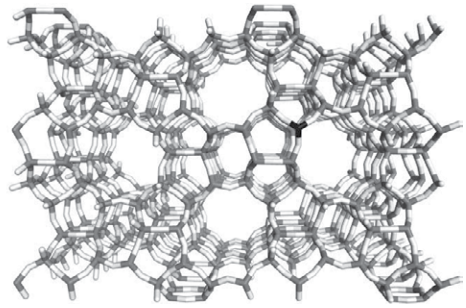


**Figure 9.** Structure of TS-1, Si (grey) and Ti (black) tetrahedral connected via oxygen bridges (white).[8]

### Hydrogen peroxide (HP-PO) process conditions

- Epoxidation conditions: < 90 °C and up to 30 bar[2–4]
- Methanol solvent is purified and recycled[3,4,6]
- Unreacted propene is also recovered and recycled to primary reactor after removal of $O_2$ (safety reasons)[3,4]
- Temperature must be regulated (heat exchangers) to avoid secondary reactions and runaway epoxidation[6]
- Total selectivity with respect to PO: 94–99%[2–4,6]

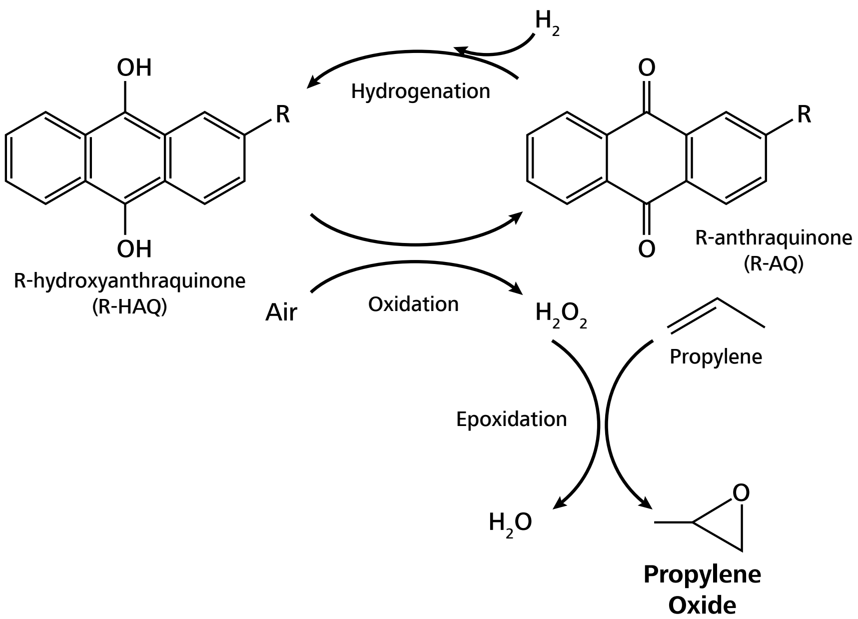



**Figure 10.** Chemical process behind the hydrogen peroxide route to produce propylene oxide.[5]

## Environmental benefits

The HP-PO process has the smallest environmental footprint compared to all other existing technologies which produce propylene oxide at an industrial scale.[4]

- Wastewater reduction: 70–80%
- Energy savings: up to 35%
- Smaller industrial footprint
- Simpler raw material integration
- No byproducts, only $H_2O$ created beside PO

**Related application note:**
Online analysis of peroxide in HPPO process: AN-PAN-1007
https://www.metrohm.com/en/applications/AN-PAN-1007

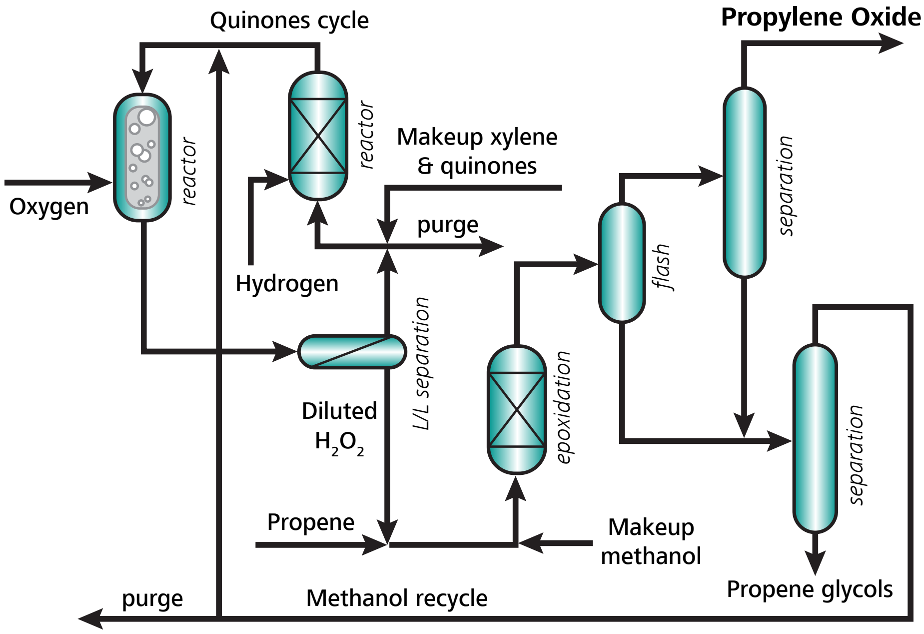
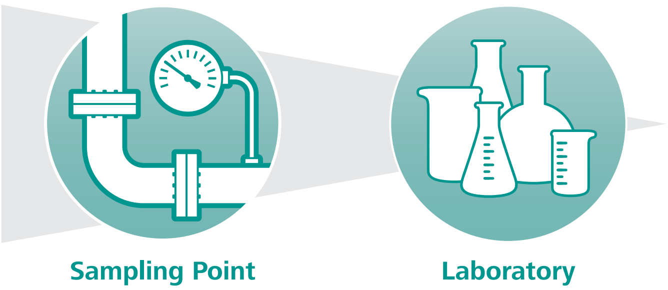



**Figure 11.** Schematic process diagram outlining the hydrogen peroxide-propylene oxide (HP-PO) method for byproduct-free production of PO. Adapted from Nijhuis et al.[2]

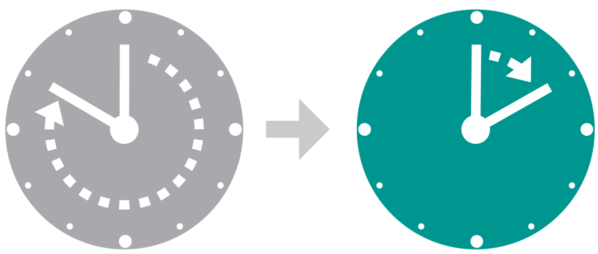
## Benefits of online process analysis

Typically, laboratory analysis for several key process parameters is the norm to keep the production facility running smoothly and safely. Manual sampling from various points along the process is a necessity, which takes up valuable time. Delays in sampling, analytical measurement, and data sharing due to these offline measurement strategies can have serious detrimental effects on production efficiency and overhead costs.




Utilization of more effective methods which increase process efficiency leads to higher productivity. Improvement of product quality can be achieved much easier with the implementation of automated process analysis, which increases profitability for manufacturers.


Time-consuming manual sampling and long distances to the laboratory are eliminated by utilizing **online**, **inline**, or **atline** process analyzers. Samples are more representative and reproducibility of results is increased as the measurements are performed exactly the same, every time. Chemical analysis performed directly at the most critical process points reduces the potential for unforeseen plant shutdowns by providing data in real-time to the central computing system.



Inline and online process analysis also tie into closed-loop control which brings production back in specification automatically with feed-forward chemical replenishment. Out-of-specification readings immediately trigger a warning at the control room, ensuring the fastest response times. Analysis occurs at the sampling point, leading to more accurate and reproducible results by the elimination of human bias and sample degradation from transport delays. Reduction of manual sampling lowers costs, saves time, and increases the safety of plant operations.


Several online and inline application solutions exist for the production of propylene oxide. Titration, photometry (colorimetry) and even reagent-free spectroscopy all play significant roles in these production processes.

Each PO production method is unique and requires a combination of analytical techniques to cover the entire process, from monitoring the raw material purity and environmental contaminants in the effluent up to and including stripping and recovery of reactants from the different process streams.

**Advantages of online analysis for PO production:**

- **24/7 analysis** for difficult to sample, hazardous substances
- Fast and **reliable** measuring techniques available (including reagent-free spectroscopic methodology)
- **No manual intervention** necessary for analysis
- **Protection** of company assets with built in **alarms** at specified warning limits, less downtime
- **Safer** working environment for employees: no manual sampling necessary ($Cl_2$, exothermic epoxidation, high temperature/pressure, autopolymerization, ATEX)
- Saving of chemicals and labor with **faster response** times
- Byproduct storage **control** options (e.g. TBC in Styrene)
- **Tighter specifications** for impurities in recycled reagents
- Increased product yield with an optimized production process: **more profitability**
- Accurate, **real-time** moisture analysis in hygroscopic sample matrix
- Purity analysis in final product made **easier**: higher quality

## Online and inline process application solutions for the production of propylene oxide

There are numerous applications for this sector which can be elevated from time-consuming manual techniques to automated online or inline process analysis solutions. A selection of these applications for the production of PO is described below.

### HP-PO: High concentration $H_2O_2$ in effluent of the primary reactor (reaction outlet line)

In the HP-PO process, hydrogen peroxide present in a methanol solvent is used as the sole oxidizing agent and is the critical feedstock and parameter to measure the complete conversion rate to PO. Thus the high demand for accurate and robust online process monitoring throughout the whole reaction process.

Hydrogen peroxide can be accurately monitored in the effluent of the primary reactor using an online analysis solution designed for extremely hazardous areas. Explosion-proof (ATEX) process analyzers are especially suitable—compliant with all electrical safety requirements, and specifically designed for high throughput processing in a hazardous industrial environment. Strict safety precautions must be implemented with all production and process equipment.

Measuring the $H_2O_2$ concentrations in the primary reaction tank plays a vital role to ensure high PO yields throughout the conversion process while reducing costs with low feedstock consumption. This application can be performed online with photometric titration. With 24/7 automated online analysis, alarms for out-of-specification values can be sent directly to the control room for fast response.

**Related application note:**
Online analysis of peroxide in HPPO process: AN-PAN-1007
https://www.metrohm.com/en/applications/AN-PAN-1007

### HP-PO: Hydrogen peroxide in the finishing reactor (upstream of propene recovery section)

Analyzing the residual $H_2O_2$ concentrations in finishing reactor overheads upstream of the propene recovery section ensures that unreacted hydrogen peroxide is closely monitored for control measures after the epoxidation reactor.

Considering the dangerous nature of this area of the process, online measurement techniques are key. Hydrogen peroxide is present in low concentrations in the finishing reactor overheads and must be measured accurately. The measurement is performed online with photometric titration, with all data sent immediately to the control room for immediate process adjustment measures in case of higher $H_2O_2$ concentrations.

**Related application note:**
Online analysis of peroxide in HPPO process: AN-PAN-1007
https://www.metrohm.com/en/applications/AN-PAN-1007

### Primary Circulation: Measuring base in the primary circulation tank

Caustic (sodium hydroxide, NaOH) is necessary in several processes which manufacture propylene oxide, especially in the chlorohydrin (CH-PO) process. The concentration of NaOH is important to monitor in recirculating caustic streams feeding the primary circulation tank.

Analysis of sodium hydroxide content is easily performed online with an industrial process analyzer configured for automated titration. With a wide range of caustic concentrations able to be accurately measured, the recirculating feed streams can be quickly adjusted to reach optimal levels.

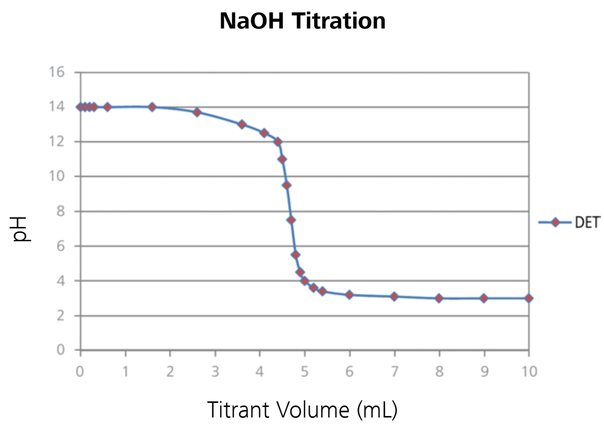



**Figure 12.** Titration curve for the determination of caustic soda (NaOH) in a chemical manufacturing process. Data provided by a wet chemical process analyzer from Metrohm Process Analytics.

### SM-PO: Monitoring TBC levels accurately in the styrene storage tank according to ASTM D4590

In the SM-PO process, styrene is manufactured along with propylene oxide. Styrene is a monomer, which polymerizes to form polystyrene, used in a wide array of industrial and consumer goods. The stabilizer 4-*tert*-butylcatechol (TBC) plays a crucial role in preventing premature polymerization during storage and transport of styrene, butadiene, vinyl acetate, and other reactive monomers.

TBC is a free radical inhibitor which requires oxygen to prevent the monomers from polymerizing. In the presence of the correct amount of TBC, peroxide radicals are scavenged. Otherwise, the peroxide radicals react with styrene monomers to form peroxide chains (polyperoxides) until the oxygen is completely depleted.

These radical species are especially hazardous during purification processes (distillation) due to the instability of peroxides at increased temperatures. In order not to compromise the product quality, the TBC concentration in styrene must stay above 10–15 mg/L. To control TBC depletion and ensure optimal storage conditions, close monitoring of its concentration is required.

**ASTM D4590** describes the specifications required to accurately measure TBC inhibitor in styrene monomer within this range, using colorimetry.[9] Automated online photometric analysis of TBC is possible 24/7 with an explosion-proof industrial process analyzer. Accurate data is provided around the clock, warning operators immediately if TBC levels are too low.

**Related application note:**
Monitoring of 4-tert-butylcatechol in styrene in accordance with ASTM D4590: AN-PAN-1027
https://www.metrohm.com/en/applications/AN-PAN-1027

### CU-PO: Determination of hydroquinone and hydrogen peroxide content in cumene production

During the hydrogenation process when producing cumene from cumyl alcohol (CMA, **Figure 7**), the oxidized stream returning to the hydrogenator, as well as the reduced stream exiting the hydrogenator, must be monitored for both hydroquinone and hydrogen peroxide content. These impurities can be detrimental to the production process for PO, affecting product quality and yield.

Performing this measurement online with titration in an industrial process analyzer allows the control room to monitor the long-term trends, optimize the process, and keep a close watch on concentrations of these chemicals outside of their programmed warning levels.

### Cumene: sulfuric acid in acetone & phenol (cleavage reactor)

The CU-PO process requires cumene as a reactant. Cumene is produced from benzene and propylene, and can be used as an intermediate in the production of other basic chemicals. Phenol, one of the products from the cumene production process, is a precursor for the production of bisphenol A (65%) which is used to make polycarbonates. Other products are phenolic resins and cyclohexanol. The production process has three stages:

- production of cumene from benzene and propylene
- conversion of cumene to cumene hydroperoxide (same process as in CH-PO)
- decomposition of cumene hydroperoxide to phenol and acetone

In this last stage, small amounts of sulfuric acid ($H_2SO_4$) are used to catalyze the reaction. Since the last reaction is very unstable, the cleavage reactor must operate under strict temperature and acidity control with a high level of acetone reflux. To prevent the formation of color bodies and other undesirable byproducts, and to minimize corrosion, it is then necessary to remove these traces of sulfuric acid prior to downstream distillation and purification. Therefore accurate and timely measurement of sulfuric acid plays an important role in cumene production (found concurrently with the CU-PO process).

This analysis can be performed online via titration with a process analyzer for both the cleavage effluent (lower concentrations) and for downstream production stages (higher concentrations).

**Related application note:**
Determination of sulfuric acid in acetone and phenol: AN-PAN-1008
https://www.metrohm.com/en/applications/AN-PAN-1008

### Finished Product: Analysis of low-level moisture in propylene oxide

Propylene oxide is a hazardous, flammable substance and therefore must be treated with extreme caution. Measurement of moisture and other impurities in the final product (as well as along the manufacturing process at critical points) is necessary to overcome unwanted side reactions or poor yields. Manual laboratory analysis methods can be quite cumbersome and can introduce bias depending on the analyst.

The hygroscopic nature of PO necessitates inline or online analysis of water content for the most precise results. Additionally, «real-time» analysis is a requirement for high throughput PO production because this gives short response times in case of process changes or increased water content in the final product.

Fast, inline analysis of low moisture content is possible with reagentless techniques such as near-infrared spectroscopy (NIRS). Suitable NIRS process analyzers are available for use in dangerous ATEX environments with robust stainless steel flow cells.

### Polyols: Measuring OH in filter feed tank discharge

Approximately 70% of PO produced globally is used to make polyether polyols, important raw materials for polyurethane production. Hydroxyl (OH) is an important functional group and knowledge of its content is required in many intermediate and end-use products such as polyols.

Analysis of KOH in the production of polyols is necessary in order to more tightly control the process and avoid unwanted reactions from occurring. Manual sampling and analysis cannot accurately monitor hydroxyl levels on a regular, continuous basis. Data transfer is slow and in the meantime, the uncorrected production process leads to suboptimal product quality or lower yields.

Online measurement of OH concentrations is made simple using, for example, an explosion-proof (ATEX) industrial process analyzer configured for conductivity measurements. Time is saved with the elimination of manual sampling procedures, and process optimization can be achieved more quickly when precise measurements of the hydroxyl content are consistently performed.

## Summary

Propylene oxide is a key industrial product, manufactured via several different methods for use in various industries. With global production of more than 7 million tons per year, PO is a major necessity for our modern lives. End products such as polyester, polyurethane, and several types of solvents would be much more difficult to manufacture without this raw material.

PO production is achieved either with or without the creation of marketable co-products. The chlorohydrin process (CH-PO) was the first large-scale production route developed, however large volumes of wastewater are produced and utility costs are exorbitant, which has led to a decline in the popularity of this method. Styrene (SM-PO) and methyl *tert*-butyl ether / *tert*-butyl alcohol (MTBE-PO / TBA-PO) production routes have grown in popularity relative to the demand and sale price of the byproducts created during manufacture. With the growing pressure to become more environmentally friendly, derivative-free processes such as cumene (CU-PO) and hydrogen peroxide (HP-PO) have been developed and are fast growing in this sector.

Manual sampling and laboratory analysis methods are slow and can introduce human error and bias, which puts employees at risk (health and safety). Additionally, the liberated samples are no longer completely representative of the manufacturing process, as many factors differ from the process after manual sampling such as temperature or pressure.

Online and inline analysis techniques performed with robust and rugged industrial process analyzers can overcome many challenges which face every industry. There are several automated solutions currently available on the market to provide these services 24/7. When utilizing online titration, photometry, ion chromatography or even inline spectroscopic techniques such as NIR analysis, companies can increase the efficiency of their operations and reduce downtime due to unforeseen events. Results are more reliable and accurate with automation, as human error is removed from the equation. Company assets are protected through automated process control and direct data transfer for immediate adjustments in critical situations. Improvement in the safety of the employees is an added bonus through the elimination of manual sampling and analysis.

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