

# DuoCondex-Process

Waste Gas Purification and Solvent recovery by Cryogenic Condensation





The production of basic chemicals or the use of solvents in the chemical and pharmaceutical industries often results in the emission of gases and vapors. This often generates exhaust gas flows of a few 100 m<sup>3</sup>/h, which are highly loaded with volatile organic compounds (VOCs). The purification of such exhaust gas streams by absorption or adsorption leads to the shift of pollutants from the gas stream in the washing liquids or adsorbents used. The exhaust air combustion is often problematic, especially in the presence of halogenated (mainly chlorinated) substances.

Condensation processes take an environmentally friendly route. However, it is only possible to reduce emissions to the limit values required by TA-Luft if liquid nitrogen (boiling point: -196 °C) is used to cool the condensers. Only then the solvents, gasoline vapors or chlorofluorocarbons (CFCs) present in the exhaust air can practically completely condense or freeze out and thus be recovered.

For example, to recover the commonly used volatile solvent dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) from an exhaust gas stream, the temperature must be decreased to -111°C to reduce the clean gas concentration to 20 mg/m<sup>3</sup>. Since typically the raw gas loadings of such exhaust gas streams amount to several 100 g/m<sup>3</sup>, recovery rates of more than 99.99% can be achieved.

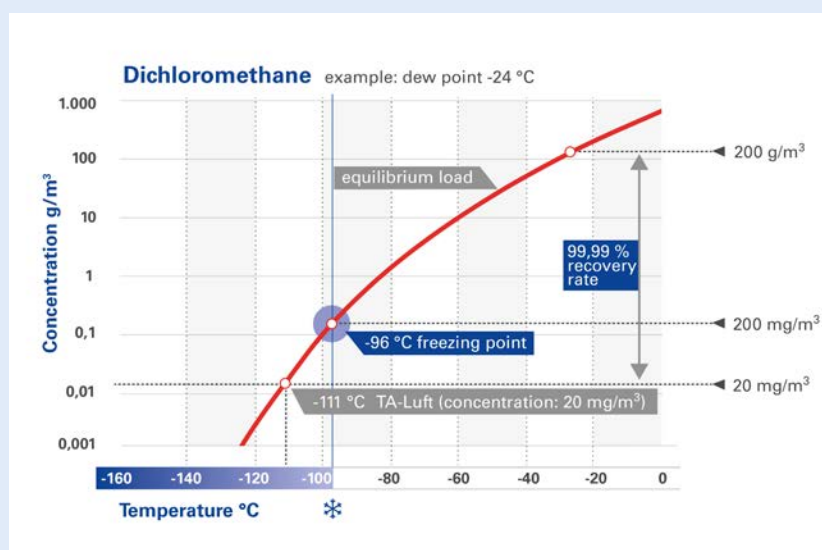


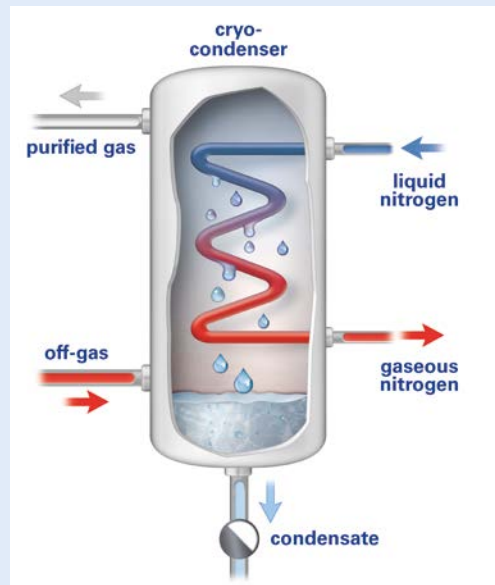
Diagram for dichloromethane

- Typical exhaust gas flows have a load of several 100 g/m<sup>3</sup>.
- To achieve a clean gas concentration of 20 mg/m<sup>3</sup>, the solvent is partially frozen (melting point: -96°C).
- For an ideal condensation process, a process gas temperature of -111°C (equilibrium temperature) is required to achieve the TA-Luft limit
- Recovery rates of more than 99.99% are achievable.

## Cryo-condenser - cooled with liquid nitrogen: good approach, problematic use

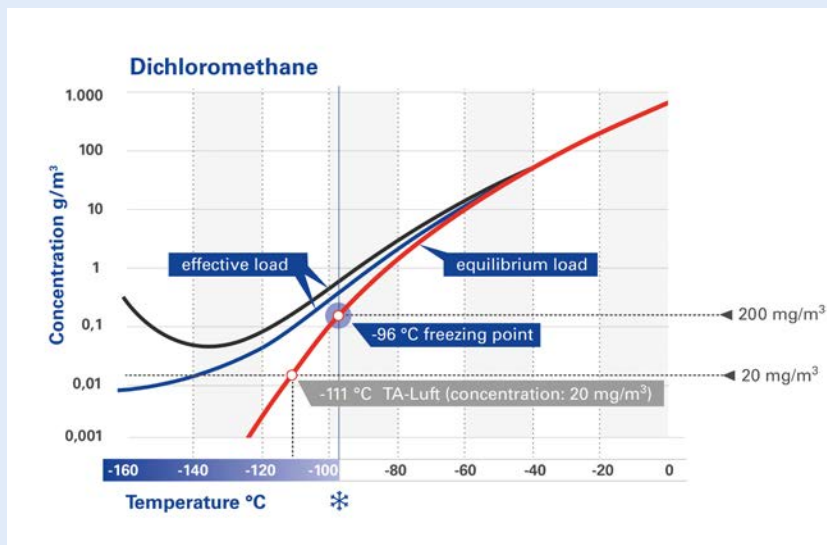
In the simplest case, heat exchangers (cryo-condensers) are used to cool the exhaust gas streams, in which the process gas is cooled in countercurrent to liquid nitrogen down to temperatures of  $-100^{\circ}\text{C}$  to  $-160^{\circ}\text{C}$ .

However, the evaporating nitrogen cools the apparatus so deep that the vapors largely freeze and quickly block the gas path. In addition, aerosols are formed. These are finest mist droplets, which are hardly deposited in the cryocondenser and therefore greatly influence the cleaning effect of the apparatus. This leads to the fact that actual process gas loading is significantly higher than one would expect based on vapor pressure calculations (equilibrium loading).



*Cryocondensation (schematic)*

- Vapor loaded exhaust gas flows through a heat exchanger (condenser).
- The condenser is cooled with liquid nitrogen.
- The vapors liquefy or they freeze on the cold heat exchanger surfaces and are thus separated from the exhaust gas.



*Load diagram for dichloromethane*

- **red:** equilibrium load (calculated on the basis of the dichloromethane vapor pressure)
- **blue:** DuoCondex process (practically achievable load)
- **black:** Increase of the process gas loading despite further lowering of the temperature if the cooling is too rapid (due to aerosol formation)

## DuoCondex process: clean and efficient

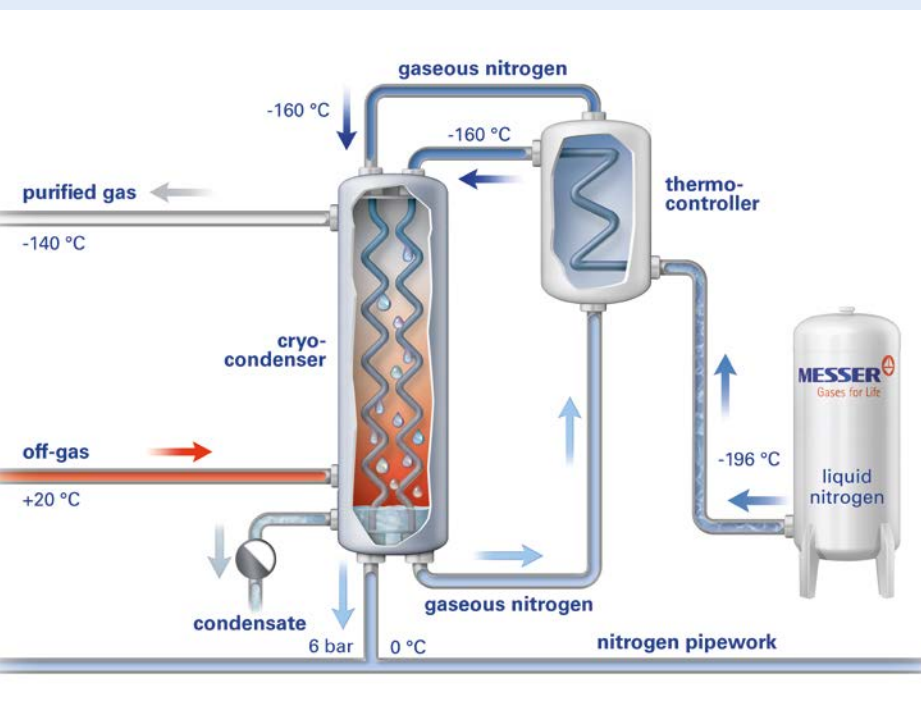
These disadvantages are avoided by the patented DuoCondex process developed by Messer. The cryogenic condenser is here not cooled by liquid, but by cold, gaseous nitrogen. As a result, the temperature difference between the process gas and the cooling medium in the interior of the heat exchanger is much lower than in the case of cooling directly with liquid nitrogen. That is the reason why the substances to be condensed mainly liquefy and barely freeze. In addition, the formation of mist (aerosols) is effectively avoided in this way and the exhaust gases can be cleaned without further treatment to the limits required by TA-Luft.

### Process Engineering: the heat exchanger arrangement makes the difference

The special heat exchanger arrangement of the DuoCondex process enables cooling of the cryo-condenser with gaseous nitrogen by full utilization of the vaporization enthalpy and the sensitive cold of the cooling medium. This is done by installation of a thermo-controller (special vaporizer).

The cryocondenser of a DuoCondex system is divided into two separate tube bundles (double condenser). The cold gas stream for cooling is provided via the upstream thermo-controller. In this apparatus, the liquid nitrogen evaporates (without warming up) and is supplied to the first bundle of the cryocondenser. The heat required for evaporation receives the thermo-controller from the cooling gas of the first tube bundle which is warmed up in countercurrent flow from the process gas. Since the available heat is about the same as the enthalpy of vaporization of the nitrogen, the gas stream from the first tube bundle can be cooled almost to the nitrogen boiling temperature and then fed to the second tube bundle.

In a third tube bundle, the cold of the purified exhaust gas can be used. This tube bundle is arranged parallel to the two nitrogen tube bundles and recuperates the cold from the purified process gas. As a result, the liquid nitrogen consumption of the process is significantly reduced and the condensation process can be further improved.



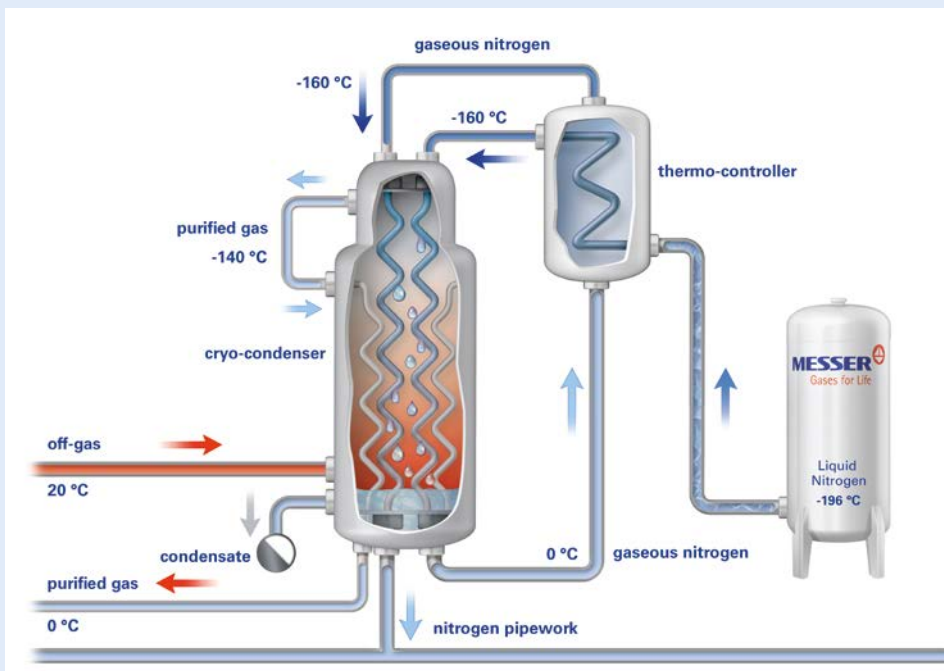
*DuoCondex process (basic principle)*

- The installation of 2 separate tube bundles in one apparatus (double condenser) combines a thermodynamic parallel flow on the process gas side with a serial flow for the cooling nitrogen.
- From every kg of liquid nitrogen, 2 kg of cold gaseous nitrogen are generated without loss, to cool the apparatus.



*DuoCondex process with cold recovery from the clean gas*

- In a third tube bundle, the valuable cold of the purified gas stream is ideally recuperated.
- The liquid nitrogen demand is significantly reduced and the condensation process approaches the theoretical optimum.



## DuoCondex systems: individually and economically

DuoCondex systems are offered as individual solutions. Special emphasis is placed on the cost-effectiveness of the installations. Therefore, the units are usually equipped with recuperators in which the cold of the purified gas stream is recovered. This leads to a significant reduction of liquid nitrogen requirements. In most cases, liquid nitrogen for cooling can after evaporation in the DuoCondex units be fed as gas into an inert gas network and thus be used twice. By appropriate design of the recuperators, the operating costs can then be minimized and the investment costs optimized.

In many cases, the condensed substances are recycled directly to the production process. This leads to further cost savings.

### Options: as varied as your claims

For particularly high demands on clean gas concentrations, we integrate an additional freezer into the process gas path. Thus, even very low-boiling substances (for example CFCs - fluorine chlorine hydrocarbons) or even gaseous substances can be removed from the exhaust gas.

Even at ideal process conditions, ice formation in the cryogenic condensers can normally not be avoided completely. In particular, when there is water vapor (humidity) in the exhaust gas, so much ice builds up in the condensers that they have to be defrosted daily (for example, overnight). For a full-continuous operation then double systems are required. While one unit is working in cleaning mode, the other unit is defrosted.

We take these and many other options into account when designing your projects.



*DuoCondex double unit*

- Due to ice formation in the condensers, cryocondensation units normally have to be defrosted daily. For a full-continuous operation then double systems are required. While one unit is working in cleaning mode, the other unit is defrosted.
- The photo shows a double DuoCondex system with a pre-condenser integrated into the process gas paths to reduce the water vapor load for the cryo-condensers.

## Special cases: e.g. recovery of methyl chloride ( $\text{CH}_3\text{Cl}$ )

Often, exhaust gas cleaning tasks are so complex that a combination of different processes or highly customized solutions are required. For example, the recovery of the gaseous methyl chloride ( $\text{CH}_3\text{Cl}$ ) affords high condensation capacities at a temperature near its boiling point. Also you have to take into consideration that this substance forms a lot of ice because of its very high vapor pressure at its melting point ( $-98^\circ\text{C}$ ). In addition, it must be made sure that the substance

does not re-evaporate after its condensation, which easily happens because of its low boiling temperature ( $-24^\circ\text{C}$ ). In the engineering of such a system it is necessary, not only to have a good design for the condensation process but also to take care about the condensate handling and the lossless recycling of the condensed gas to the production process or to the disposal system.



*Recovery unit for methyl chloride*

- The plant has a condensation capacity of 2,000 kg/h  $\text{CH}_3\text{Cl}$  (batch operation).
- Downstream freezers (operation temperature:  $-170^\circ\text{C}$ ) reduce the residual  $\text{CH}_3\text{Cl}$  - load to TA-Luft limits.
- Two cooled storage tanks allow lossless recycling of

- the liquefied solvent to the production process.
- The plant can be used to evacuate the reaction vessels so that no unwanted methyl chloride emissions are produced in the production plant.



The system can also be operated under reduced pressure on the process gas side. As a result, the connected vessels can be evacuated and no unwanted methyl chloride emissions are generated in the customer's production facility.

## Pilot plant: test DuoCondex

The practical test is always the best way to prove the performance. We will demonstrate the process on site with our mobile pilot plant designed for a gas flow of 200 m³/h. Under real operating conditions, the data required for a detailed individual engineering can be determined most reliably

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*Pilot plant for customer testings*

### Your benefits at a glance

- Exhaust gas purification with simultaneous recovery of the solvents
- Cost savings through recycling of solvents
- Compliance with TA-Luft limits
- Full use of the latent and sensitive cold of liquid nitrogen
- Dual use of liquid nitrogen: Nitrogen that evaporates in the process can afterwards be used for inerting.
- Individual system design with many options
- Pilot plant available for trials in your facility



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