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Gas applications in fine and specialty chemistry.

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Contents.

Introduction.

Fine and specialty chemistry is an important part of the chemical industry. Products include complex mixtures as well as pure chemical substances. Most of these are manufactured in multi-product plants using stirred-tank reactors, often with several process steps combined in one reactor.

The worldwide market for fine chemicals is estimated at US$ 75 billion [1]. Major product groups include pharmaceuticals, adhesives, pesticides, catalysts and enzymes, dyes and pigments, chemicals for the electronics industry, flavours and fragrances, food and feed additives and special polymers (see fig. 1).

Facilities for manufacturing such products have capacities between a few hundred and 10,000 tonnes per year. Table 1 describes selected products by industry, nature of reaction, scale of production and type of reactor [3].

The following are the main process steps in the manufacture of fine and specialty chemicals:

- Storage and conditioning of feedstocks (e.g. size reduction, weighing, metering)
- Synthesis
  - Chemical (e.g. hydrogenation)
  - Biotechnological (e.g. fermentation)
- Separation and purification (e.g. distillation, extraction, crystallisation, centrifugation)
- Drying
- Product conditioning (e.g. pelletising)
- Storage and packaging

Fig. 1: Survey of product groups in fine and specialty chemistry [2]

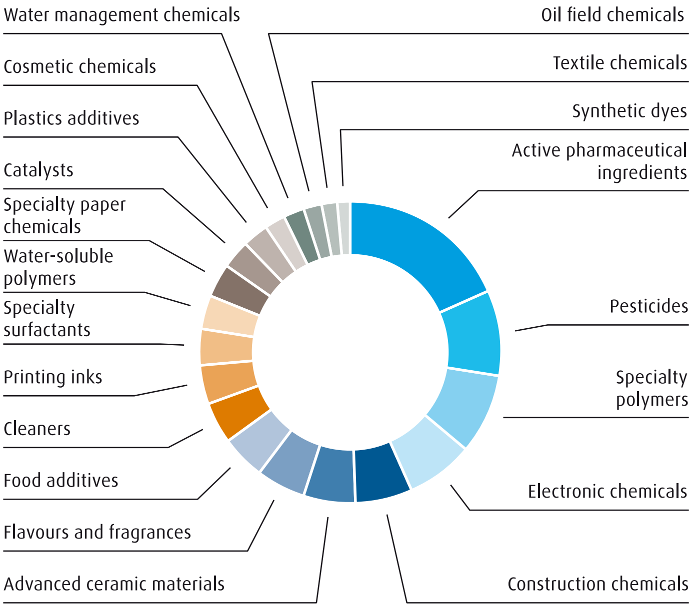


Table 1: Selected fine chemical products [3]

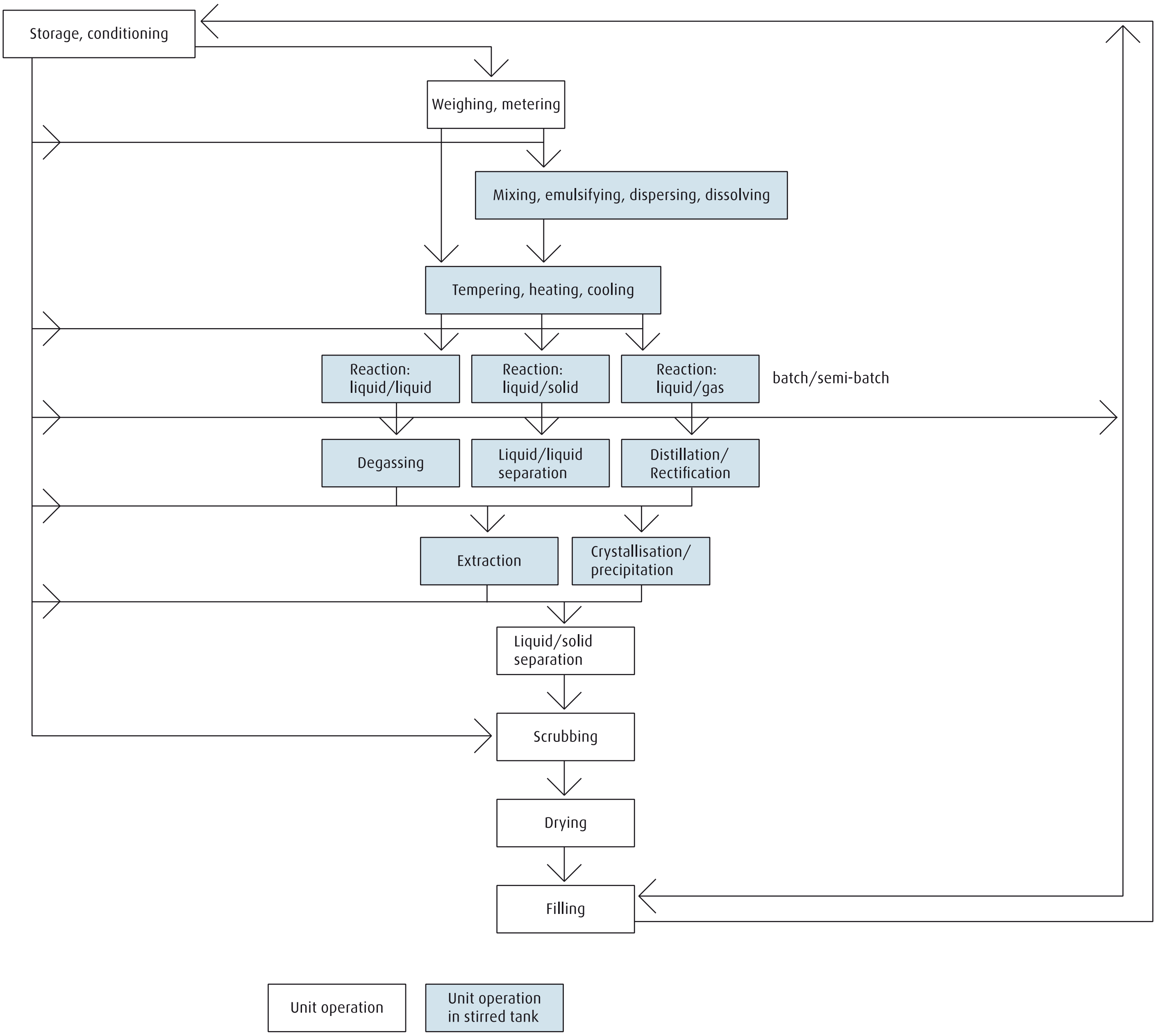
No.	Product	Nature of Industries	Catalytic (C)/ Non-Catalytic (NC)[a]	Scale of Operation (tpa)	Type of Reactor[b]
1	Trimethylhydroquinone; Isophytol; Vitamin E	Vitamin	C (Homo; Het)	500–1000	STR
2	Methyl heptenone	Pharma; Aroma	NC & C (Het)	1000–2000	CR (nozzle)
3	Vitamin A (Wittig reaction)	Vitamin	NC	1000	CR (nozzle)
4	Isobutylbenzene Ibuprofen	Pharma Nonsteroidal analgestic	C (Homo; Het)	1000–3000	STR
5	Fenvalerate *p*-Hydroxy benzaldehyde	Agrochemical	C (PTC)	300–500	STR
6	*p*-Anisic aldehyde	Pharma; Aroma and Flavour; Agrochemical	C (Homo; Het)	1000–3000	STR; EC
7	Catechol; Hydroquinone	Agrochemical; Aroma and Flavour; Photography; Additives (antioxidants)	C (Homo) & NC	1000–5000	STR; BCR
8	p-Amino phenol	Pharma	C (Het) & NC	1000–5000	STR; EC
9	Isocyanates[c]	Pharma; Agrochemical; Rubber	NC	300–2000	STR
10	Citral	Aroma; Pharma	C (Het)	1000–3000	CR (short bed)
11	2,6-di-*tert*-butylphenol	Additives (antioxidants)	C (Homo)	1000–2000	STR
12	Phenylglycine/ p-hydroxyphenyl glycine	Pharma	NC & C (Bio)	1500–2000	STR; CR
13	*p*-*tert*-butylbenzaldehyde; Benzaldehyde/Benzyl alcohol	Aroma; Pharma	C (Homo; Het)	1000–5000	STR
14	1,4-dihydroxymethylcyclohexane	Polyester	C (Het)	5000	CR
15	Phenylethylalcohol	Aroma; Pharma	C (Homo; Het)	1000–3000	STR
16	Anthraquinone (AQ) and 2-alky AQs	Dyes; H_2O_2; Paper	C (Het)	500–3000	STR
17	Indigo	Dyes	C (Het)	300–1000	STR
18	Diphenyl ether; *m*-phenoxy toluene	Aroma; Heat Transfer Fluids; Agrochemical	C (Het)	1000–10000	CR
19	Benzyl toluenes	Heat Transfer Fluids	C (Het)	500–2000	STR
20	*o*-, *m*-, and *p*-Phenylene-diamines	Dyes; Agrochemical; Aromatic Polyamide Fibres	C (Het)	1000–3000	STR
21	2,2,6,6-tetramethylpiperidinol	Additive (Light stabiliser)	NC & C (Het)	1000–2000	CR
22	Glyoxalic acid	Pharma	NC	500–2000	STR; BCR

Notes
[a] Homogeneous (Homo); Heterogeneous (Het); Biocatalytic (Bio); Phase Transfer Catalysis (PTC).
[b] Stirred-Tank Reactor (STR); Bubble-Column Reactor (BCR); Continuous Reactor (CR); Electrochemical (EC).
[c] E. g. *n*-propyl/*n*-butyl; cyclohexyl; p-isopropylphenyl isocyanate; isophorone diisocyanate; 1,5-naphthalene diisocyanate.

A frequently taken approach to ensuring effective capacity utilisation and high flexibility in terms of substances, technologies and conditions is the use of multi-product plants. Fig. 2 is a block diagram of a batch multi-product facility [4]. The figure makes it clear that many unit operations are carried out in a stirred-tank reactor, which makes it the most important apparatus.

While batch operation predominates in fine and specialty chemistry, continuous multi-product systems are also used where high capacity is needed. These are single-train plants which are defined by the synthesis taking place and the product classes involved. Examples are hydro-genation and chlorination reactions.

Fig. 2: Block diagram of a batch multi-product plant [4]



Technical and specialty gases find use in many synthesis processes and a number of unit operations, in analysis and in plant maintenance. For this reason, the Linde portfolio for gas applications in fine and specialty chemistry has been developed to include the following elements:

- Gases and gas mixtures for synthesis reactions
- Instrumentation and controls for inerting with nitrogen or carbon dioxide
- Inert gas locks for vessels and reactors
- Processes and apparatus for cooling reactors with liquid nitrogen as well as heating
- Processes and apparatus for treating off-gases by cryocondensation of hydrocarbons with liquid nitrogen
- Processes and apparatus for product size reduction using liquid nitrogen (cold milling, prilling)
- Service procedures for cleaning, inerting and drying apparatus as well as equipment for cleaning with CO_2 particles or CO_2 snow

Synthesis operations in fine chemistry.

Synthesis reactions are carried out in batch and continuous stirred tanks, bubble-column reactors and microreactors, as table 1 shows in part. Stitt [5] has described reactor types for the manufacture of fine chemicals and listed their advantages and disadvantages. The preferred type, the stirred-tank reactor, is operated on small and large scales, with homogeneous or heterogeneous catalysts, often with gas dispersion and with complete mixing by a variety of stirrers. A wide variety of synthesis reactions – 45 different ones just in the organic branch [1] – is performed in stirred-tank reactors.

Many syntheses involve gases or gas mixtures, some of which are referred to as specialty gases. Table 2 lists selected synthesis reactions with the gases involved. Purities are also indicated for pure gases.

Table 2: Selected synthesis reactions in fine and specialty chemistry, with gases required

Synthesis reaction	Gas	Available purities [6]
Amination	Ammonia	≥ 99.98 to 99.9999
Acetylation	Acetylene	≥ 99.6
Carbonylation	Carbon monoxide	≥ 99 to 99.997
Chlorination	Chlorine	≥ 99.8 to 99.999
Fermentation	Oxygen	Air, or oxygen-enriched air with up to 80 % oxygen
Hydrogenation	Hydrogen	≥ 99.999 to 99.99999
Oxidation	Oxygen (or air)	≥ 99.6 to 99.9999

When gases such as oxygen and acetylene are used in synthesis operations, specific equipment ensures fast and safe gas supply.

The principal use of oxygen is to boost process intensity, for example in the production of vinyl acetate from ethylene by multi-stage oxidation. An instrumentation and control unit and a gas injector are required along with an oxygen supply; the gas can be delivered in liquid form, generated on site or supplied from a pipeline. The instrumentation and control unit (fig. 3) ensures safe and reliable metering of oxygen in the various operational states of the reactor or system. This unit therefore features a "block and bleed" system, which prevents oxygen from getting into the piping to the reactor in case, for example, the air is cut off. This system also serves as the interface to the process instrumentation and control system.

The gas injector (fig. 4) is matched to the application in question. It ensures good mixing of oxygen with air. It is important that mixing takes place over a short distance, that the mixture is as homogeneous as possible and that the oxygen does not impinge directly on a pipe wall. Instead of a gas injector in the process air piping, a gas distributor can be placed in the reactor if it is desired to meter oxygen directly into the reactor or the reactants inside it.

Fig. 3: FLOWTRAIN® FT500 instrumentation and control unit for safe oxygen metering (capacity 50–500 Nm^3/h)

Fig. 4: OXYMIX™ oxygen injector for oxygen enrichment

Fig. 5: LINDOMATIK® pressure regulating and safety equipment

Today, acetylene is used chiefly in processes of specialty and fine chemistry, such as the manufacture of vitamins, vinyl ether, fragrances, plastics additives and special plastics. Mobile acetylene delivery units (16-cylinder bundle, 8-bundle and 16-bundle trailers) in conjunction with on-site pressure control and safety equipment (fig. 5) make it possible to supply acetylene quickly and safely to a multi-product plant.

Biotechnological processes are going to be increasingly important in fine and specialty chemistry. Many vitamins, amino acids, aromas, biopolymers and acids are already being manufactured by this route. Industrial gases are also required for the processes involved. Some aerobic fermentations are carried out with oxygen-enriched air. Nitrogen is used as a stripping gas, as an inert gas in the processing of flammable solvents and in the product quality assurance effort. It also serves for the conveying of liquids and solids in process systems. Liquid nitrogen is furthermore an effective coolant whose applications include freeze drying. Carbon dioxide can function as a carbon source for autotrophic microorganisms and is also used in pH regulation. Ammonia gas is a good nitrogen source and also figures – frequently in the same pass – as a pH control agent.

Cooling and heating systems for syntheses.

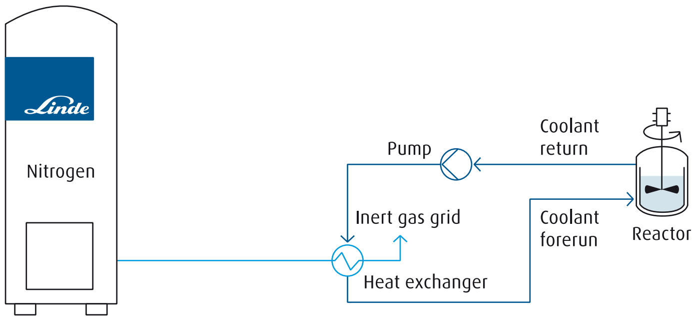
Multiple synthesis reactions or unit operations are often conducted at different temperatures in stirred-tank reactors. A reaction specific to fine and specialty chemistry is low-temperature synthesis, which can well require temperatures as low as -110 °C. Low temperatures improve selectivity and lower the costs of isolating the products. In many cases, the specified temperature must also be maintained very accurately in order to minimise the quantity of byproducts formed; temperature accuracies of ±1 °C are feasible. Liquid nitrogen is a good medium for producing low temperatures (below -40 °C) because the cooling capacity is highly flexible and investment costs are lower than those for conventional refrigeration systems. Maintenance costs are much lower as well.

Typical low-temperature processes [7] are:
- → Use of organolithium compounds
- → Asymmetric syntheses
- → Birch-Hückel reduction
- → Grignard syntheses
- → Reduction of metal hydrides
- → Wittig reaction
- → Low-temperature crystallisation

A variety of cooling methods are in use:
- → Injecting liquid nitrogen to cool the reaction mass directly
- → Injecting liquid nitrogen into an integral heat exchanger or cooling jacket to cool the reactor contents
- → Cooling and/or heating a reactor via a secondary loop carrying a heat/cold transport medium cooled or heated in heat exchangers; liquid nitrogen is generally employed for cooling

Special systems have been developed for cooling; fig. 6 is a schematic diagram of a simple one.

Fig. 6: Schematic diagram of a simple cooling system



Standardised units in the CUMULUS® series have been designed and built for cooling capacities of 5, 20 and 50 kW. Fig. 7 shows the CUMULUS® systems, which can also be easily installed in laboratory or pilot plants.

Fig. 7: CUMULUS® standard cooling systems

Because reactor temperature control requirements are stringent – particularly in terms of broad temperature range and rate, and accuracy of temperature adjustment – cooling and heating systems [8] have been developed.

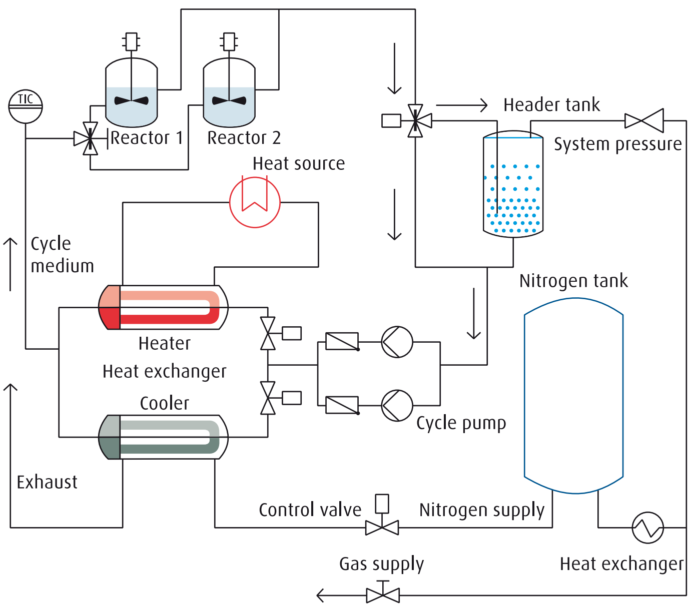
Fig. 8 presents a typical scheme for a CRYOHEAT® system. The main elements are the heat exchangers for cooling and heating the heat transfer medium, one or two pumps to circulate it, an expansion vessel, and valves that permit rapid and reliable temperature adjustment. The medium is cooled against liquid nitrogen and can be heated with electricity, steam or another heat loop. Technical implementation of such a broad temperature range also calls for a suitable heat transfer medium such as methylcyclopentane [9] or a well-defined mixture of hydrocarbons [10].

Fig. 9 shows an example of a pilot plant installation for reactor temperature control. The key parameters are:

Key parameters

Temperature range	-110 to +130 °C
Pressure range	Up to 10 bar
Cooling capacity	50 kW
Heating capacity	56 kW

Fig. 8: Schematic diagram of the CRYOHEAT® cooling and heating system



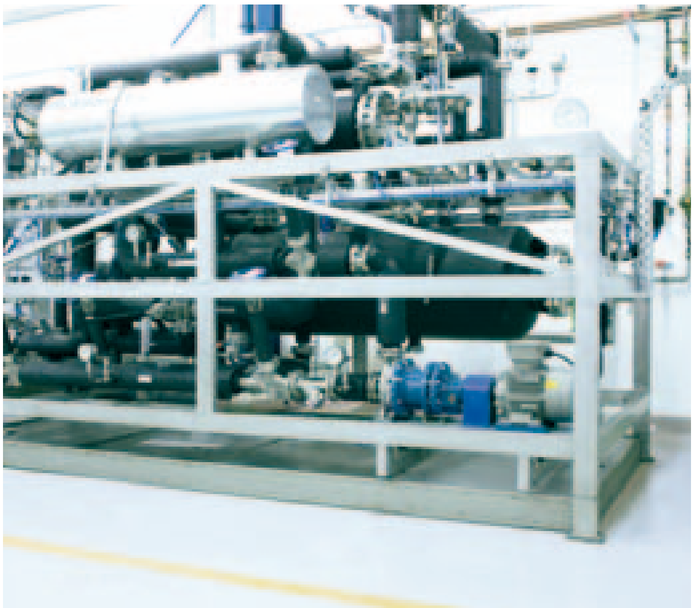


Fig. 9: CRYOHEAT® cooling and heating system

Inerting.

Inerting uses an inert gas (usually nitrogen but also carbon dioxide in some cases) to displace atmospheric oxygen, combustible gases and moisture. Inerting is employed for safety reasons or to protect products. Safety concerns include:

- Safe startup and shutdown of apparatus
- Prevention of an explosive atmosphere
- Avoidance of explosion hazards when handling combustible fluids

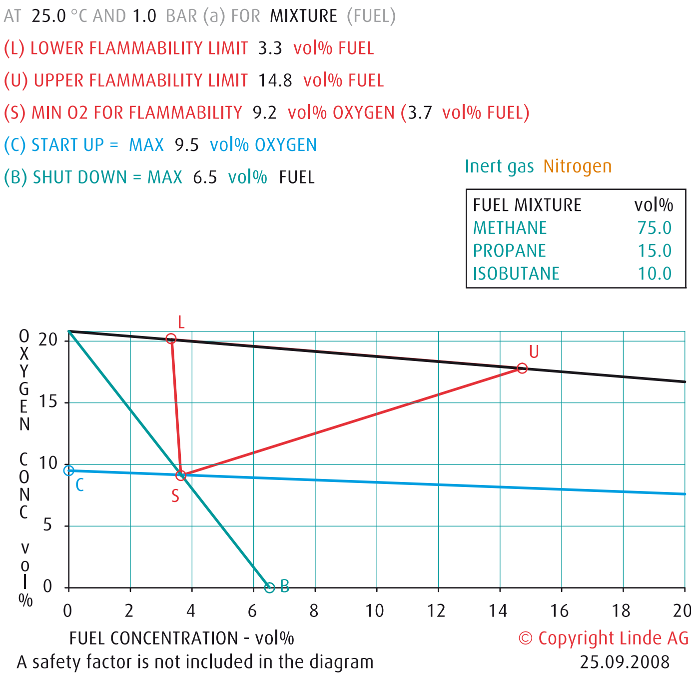
Products are protected by suppressing oxidation reactions with atmospheric oxygen and/or blocking the access of moisture.

The following techniques are used in inerting:

- Dilution purging
- Displacement purging
- Pressure swing purging (pressure rise, pressure relief or vacuum)
- Blanketing

Knowledge of the explosion limits is essential for many processes and operations in fine and specialty chemistry. The explosion limits depend on the pressure and temperature as well as on the composition of the mixture present. Along with experimental determinations, software for calculating the explosion limits is gaining in importance; an example is the safety system created by Linde Gas [11]. Fig. 10 shows a calculated explosion triangle. Another program computes in advance the time and the quantity of nitrogen required for various inerting techniques.

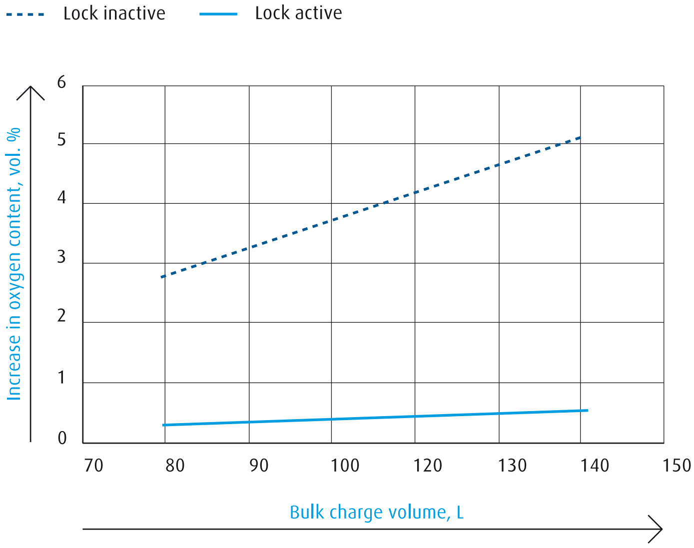
Fig. 10: Safety system – safety triangle (screenshot)



Fine and specialty chemical processes involve not only liquids and gases but also solids, for example in reactions carried out in stirred tanks. In order to ensure safe handling of solids and prevent side reactions, such as oxidation, inert gas locks [12] have been devised to block the entry of oxygen when solids are being charged into reactors and vessels. These fittings also prevent emissions and protect against moisture and electrostatic charge. Fig. 11 shows an example of an inert gas lock.

Fig. 11: N2LOCK® inert gas lock integrated into the charging port of a mixer

Fig. 12: Change in O_2 concentration in vessel versus bulk charge volume with and without active lock



Among the features of inert gas locks are:
- Minimal access of oxygen to the vessel during opening and charging
- Low nitrogen consumption
- Inexpensive integration into charging ports of existing vessels
- Easy day-to-day handling
- Variety of designs for particular applications

Fig. 12 is a plot of the oxygen concentration versus the charge volume for a vessel being charged with and without an inert gas lock. The use of the lock makes it possible to cut the rise in the oxygen level from between 2.9 and 5 vol. % to about 0.3–0.6 vol. %.

Locks, therefore, are a simple but effective way of ensuring very slight access of oxygen and moisture to a vessel or reactor when it is charged with solids.

Cryocondensation.

Cryocondensation means cooling off-gas streams against liquid nitrogen in heat exchangers until the valuable or noxious substances contained in them condense or freeze onto the heat transfer surfaces. In the process, the liquid nitrogen is vaporised and becomes available for further use, such as in inerting.

The temperature necessary to get below the dewpoint or to attain compliance with regulatory limits can easily be adjusted and controlled through the use of liquid nitrogen (-196 °C at 1 bar) as the cold transfer medium. Cryocondensation thus brings within reach the temperatures required, which can be below -150 °C in some cases.

The technique is frequently employed in fine and specialty chemistry. Simple technology, high flexibility and increasingly stringent environmental regulations suggest that its use will increase.

Cryocondensation is employed mainly for the following purposes:
→ Compliance with environmental regulations, such as the TA-Luft, a German air quality directive
→ Recovery of valuable substances, for example by condensation and recycling of expensive hydrocarbons to the reactor
→ Reduction of damage to downstream apparatus, such as diminished corrosion due to removal of chlorinated hydrocarbons

Fig. 13: CRYCON® cryocondensation unit in fine chemistry

Compared to other techniques for off-gas treatment, cryocondensation offers several advantages:
→ It is environmentally safe because no secondary burdens are generated as for example in scrubbing or adsorption
→ It involves no auxiliary materials as do processes such as absorption
→ It yields low residual loads
→ The condensate can be reused directly
→ Nitrogen is used twice, for cooling and for inerting (in the plant's nitrogen grid)
→ Maintenance costs are low because the system has few moving parts
→ The process is flexible in terms of the VOC (volatile organic compounds) load
→ The unit is simple to operate and lends itself to full automation
→ Investment costs are relatively low

In comparison with the use of refrigeration equipment, liquid nitrogen makes it possible to achieve the required temperatures economically and without difficulty. In particular, the condensation power can be adjusted relatively quickly and over a broad range by controlling the rate of injection of liquid nitrogen.

Cryocondensation can be used economically for treating off-gas streams heavily loaded with hydrocarbons and for achieving very low residual loads (down to the ppm range). It finds successful use with substances such as dimethyl ether, toluene, tetrahydrofuran, ethyl acetate and acetone as well as mixtures such as dimethyl ether-methyl chloride or acetone-methanol-dichloromethane.

The ease of combining cryocondensation with other treatment and recovery processes, adsorption in particular, permits an expansion of the field of application. Throughputs of up to 1500 Nm^3/h of off-gas and very high purities can be realised economically with such a combination.

Fig. 13 shows a CRYCON® cryocondensation apparatus integrated into a fine chemicals manufacturing plant. As a rule, incorporating complete prefabricated cryocondensation equipment into existing systems is a quick and easy job. This installation has the following key parameters:

Key parameters

Off-gas capacity	80 Nm^3/h
Off-gas temperature	+20 to +25 °C
Max. operating pressure	16 bar (raw gas and nitrogen side)
Transport gas	Nitrogen
Condensation temperature	-90 to -110 °C
Coolant	Liquid nitrogen
Performance	Below TA-Luft limits

After cryocondensation, the nitrogen gas is delivered to the plant's nitrogen grid, while the recovered hydrocarbons are recycled.

Fig. 15: The CIRRUS® series of standard VEC (vapour emission control) units

Another application of cryocondensation is fractionating condensation [13], in which different temperature levels are realised in each individual heat exchanger. Test units are available for such processes (fig. 14).

Hydrocarbon recovery from off-gases has both environmental and economic benefits. On the one hand, pollution is reduced by averting the production of CO and CO_2, especially if the off-gases are burned. On the other hand, plant investments for the recovery of valuable hydrocarbons by cryocondensation can pay for themselves within 0.5–2 years, depending on prices and other conditions.

Moreover, standardised cryocondensation systems have been developed in the CIRRUS® series. These units have process gas stream capacities of 50, 150 and 500 Nm^3/h and operate at moderate pressures. Fig. 15 illustrates the CIRRUS® equipment. Off-the-shelf cryocondensation units have the advantages of ready availability and lower cost.


Fig. 14: Test unit for hydrocarbon recovery by three-stage cryocondensation

Size reduction.

Many fine and specialty chemicals have to meet rigorous standards, not just on purity but also on particle form. Accordingly, size reduction processes are important, along with purification operations such as crystallisation.

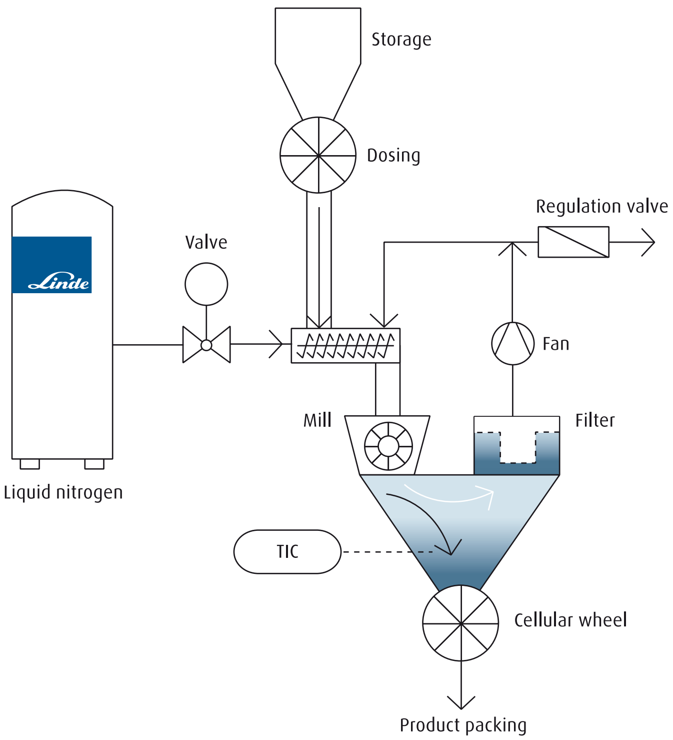
Some of these processes, such as cold milling and prilling, require industrial gases. Liquid nitrogen is employed as coolant when cold milling is performed in impact and centrifugal mills. The nitrogen supply system can be designed very quickly and adapted to a variety of requirements. Several useful effects can be achieved through the use of liquid nitrogen in milling:

- → Fine grain size
- → Free-flowing quality of product, hence good conveying properties
- → Protection against fire and dust explosions by inert nitrogen
- → Enhanced mill capacity

A technical example of a cold milling system is illustrated in fig. 16. The screw conveyor in the schematic diagram transports, meters and cools the milling feed with liquid nitrogen.

A further option for cooling and inerting is direct metering of liquid nitrogen into the mill.

Fig. 16: Cold milling system made up of a Linde screw conveyor-cooler and a hammer mill



Industrial services.

Services to the fine and specialty chemicals industry include a wide range of processes and hardware, some of them making use of gases. These services help reduce maintenance time and costs, ensure and enhance workplace safety and abate environmental pollution. Another important point is that the gases used, such as nitrogen, are non-toxic, incombustible and inert.

The following principal operations are encountered in industrial service:
- → Purging and drying of plants with nitrogen
- → Cleaning of units and piping
- → Leak testing with N_2/He mixtures

In addition, carbon dioxide is increasingly used for cleaning, as in the CRYOCLEAN® process [14], where surfaces to be cleaned are blasted with high-velocity CO_2 (dry ice) pellets. The process lends itself to cleaning not only freely accessible surfaces, but piping as well.

The advantages of the process are that it requires no solvents, the blasting medium leaves no residues behind and no wastewater is generated.

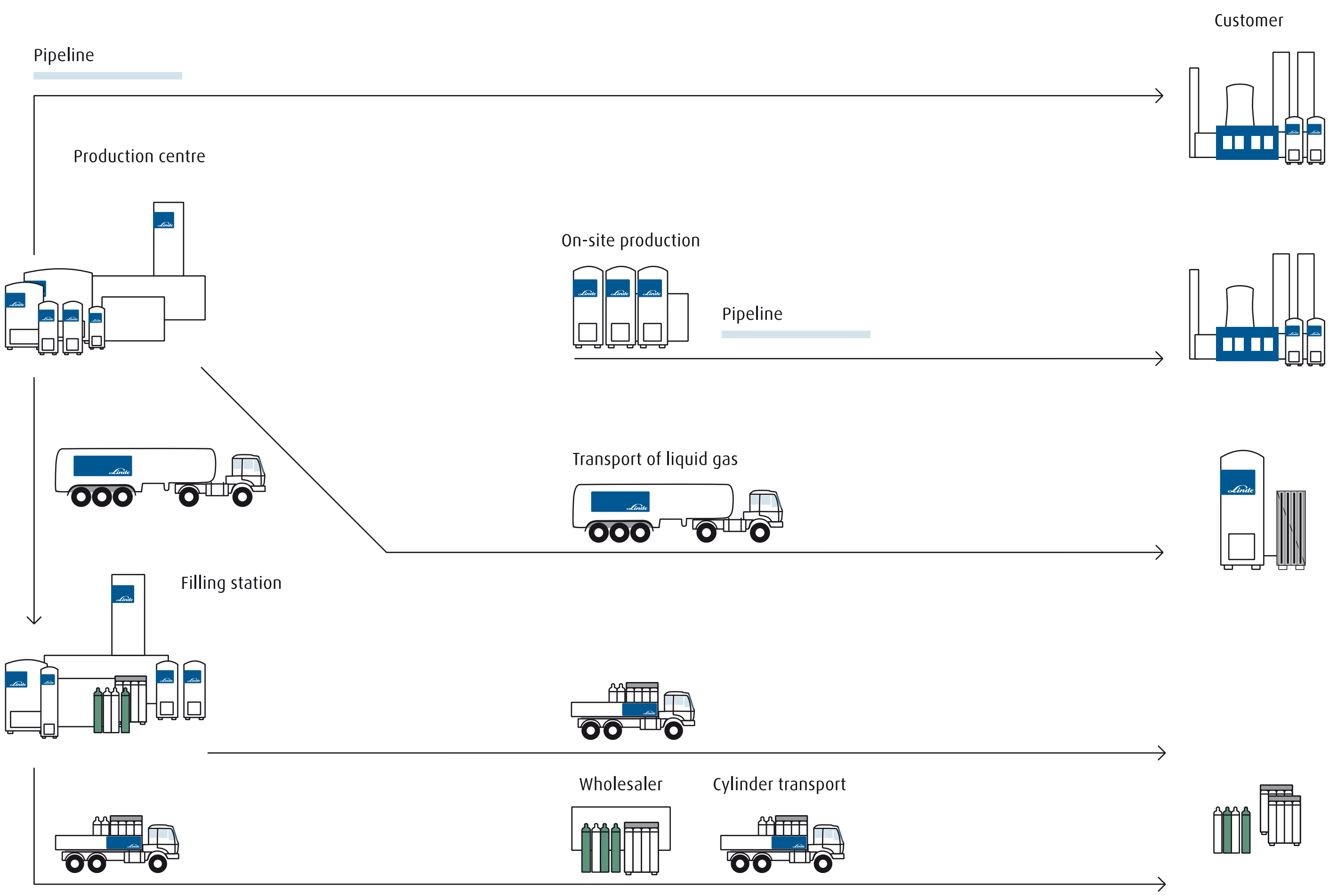
Fig. 17: Cleaning with the CRYOCLEAN® process in a coffee processing plant

Gas supply.

Gas demand in fine and specialty chemistry varies widely in terms of both quantity and gases used. Gases can be delivered in small containers, standard cylinders, cylinder bundles, steel drums, battery vehicles or bulk tank vehicles, or they can be generated on site. Fig. 18 illustrates the basic relationships governing supply options.

The most frequently used gases are nitrogen, oxygen and hydrogen. Along with other industrial gases, such as carbon monoxide, synthesis gas and carbon dioxide, there is also a need for inorganic and organic gases at various purities as well as for gas mixtures. Specialty gas catalogues [6] provide further details. Moreover, the LIPROTECT™ platform supports the safe handling of gases. It includes training, service (total gas management) and special products to enhance safety, such as odorants for certain gases.

Fig. 18: Survey of delivery options and their relationships



Outlook.

The field of fine and specialty chemicals is expected to grow on average by 6 % annually in the coming years [1]. Demand in biotechnological processes will grow even faster, 10–15 % per year.

Future market development will be shaped, above all, by globalisation: Asia, especially China and India, will produce more and more fine and specialty chemical products. Product quality and environmental standards in these countries will naturally become stricter as well. One implication is that more effort must be put into off-gas and wastewater treatment in order to meet emission limits. This means greater demand for gases in these countries.

The U.S. and Europe will increasingly concentrate on the manufacture of end products and high-value specialty products. In this context, special synthesis operations, such as low-temperature syntheses, may become more important in these markets.

Research and development will play an increasingly vital part in retaining production sites in the leading industrialised economies. Accordingly, product development – e. g. in nanoparticles – and an intensified search for new technologies and broader applications for these processes will be pushed. New openings for the use of technical and specialty gases in fine and specialty chemistry will appear as a result.

Summary.

This publication surveys applications for gases in fine and specialty chemistry, ranging from synthesis processes and inerting to off-gas treatment and industrial services. Processes and their fields of use are briefly explained, and existing technical solutions are presented to illustrate the hardware involved. Finally, options for gas supply are summarised and forecasts are made for future developments in this field.

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For additional info on gas uses in chemical industries, please refer to: Bernhard Schreiner, Hans-Jürgen Reinhardt: "Outlook for industrial gases in the petrochemical industry," Hydrocarbon Processing, December 2008, 69–80.

Getting ahead through innovation.

With its innovative concepts, Linde is playing a pioneering role in the global market. As a technology leader, it is our task to constantly raise the bar. Traditionally driven by entrepreneurship, we are working steadily on new high-quality products and innovative processes.

Linde offers more. We create added value, clearly discernible competitive advantages, and greater profitability.
Each concept is tailored specifically to meet our customers' requirements – offering standardised as well as customised solutions. This applies to all industries and all companies regardless of their size.

If you want to keep pace with tomorrow's competition, you need a partner by your side for whom top quality, process optimisation, and enhanced productivity are part of daily business. However, we define partnership not merely as being there for you but being with you. After all, joint activities form the core of commercial success.

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