ENVIRONMENTAL
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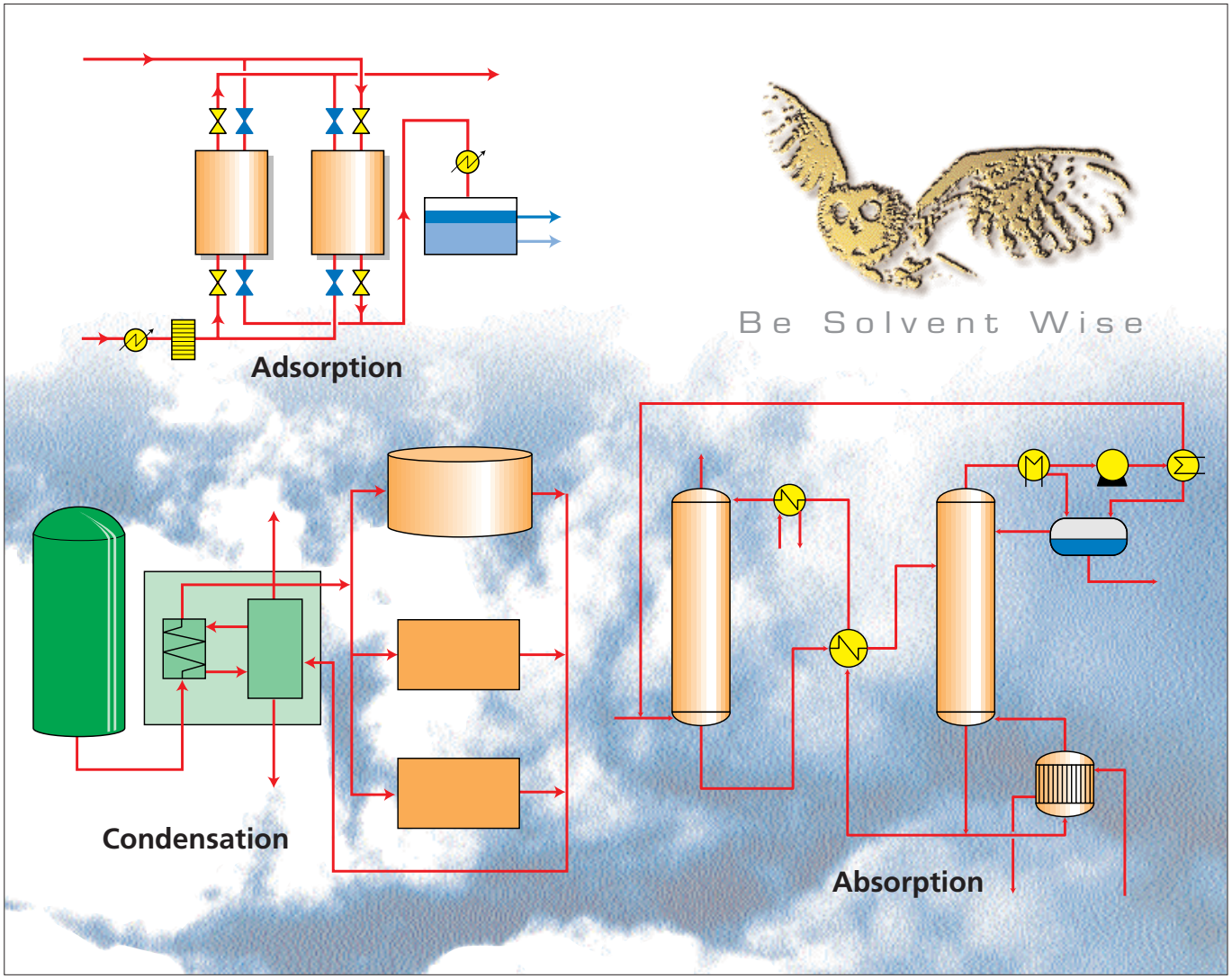
PROGRAMME

GG12
GUIDE

SOLVENT CAPTURE FOR RECOVERY AND RE-USE FROM SOLVENT-LADEN GAS STREAMS



GOOD PRACTICE: Proven technology and techniques for profitable environmental improvement

SOLVENT CAPTURE FOR RECOVERY AND RE-USE FROM SOLVENT-LADEN GAS STREAMS

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SUMMARY

This Good Practice Guide provides detailed information about proven technologies and techniques for the capture, recovery, and subsequent re-use of organic solvents from solvent-laden gas streams. It is intended to help companies consider fully if solvent capture and re-use could be cost effective for them and, if so, to select the most appropriate technology for the recovery of solvent from their process emissions.

Different categories of the three main solvent recovery techniques - adsorption, condensation and absorption (scrubbing) - are considered. For each technology, the Guide discusses:

- general principles;
- applicability;
- advantages and disadvantages;
- equipment operation and installation;
- utility requirements;
- factors affecting capital and operating costs.

In addition, the Guide considers new and emerging technologies such as membrane processes.

The Guide stresses that the best choice is strongly dependent on the nature of the air stream, the properties of the solvent concerned, and the specific application. The Guide presents information on measures to improve the cost-effectiveness of solvent capture and re-use. For example, re-using the captured solvent, preferably directly, makes recovery much more attractive economically, and lowering the maximum volumetric airflow through a system reduces both the capital and operating costs of recovery equipment. An indication of the range of flow rates and solvent concentrations for each technology is given in the Guide.

Adsorption is an extensively used recovery technique. A range of adsorbents is available, with granular activated carbon (GAC) the most common choice. Desorption/recovery is carried out either by using steam or a hot inert gas, or under vacuum.

Recent developments mean that condensation using a range of coolants and refrigerants is a viable stand-alone option for solvent recovery. It is considered highly suitable for organic solvents with reasonably high boiling points and which are present in appreciable concentrations. Cryogenic condensation using liquid nitrogen is an option for solvents with low boiling points. It is particularly economic for those companies that use liquid nitrogen on site.

Absorption or scrubbing is particularly suitable for solvents that are readily soluble in water or for high boiling point organic compounds. Organic scrubbing liquids are usually regenerated by steam stripping; aqueous ones by distillation.

The last section in the Guide is intended to help highlight the issues that can determine whether or not solvent capture and re-use will be a viable option at your site.

CONTENTS

Section			Page
1	**Introduction**		1
	1.1	Target processes	1
	1.2	Making solvent capture more cost-effective	2
2	**Technology selection**		4
	2.1	Factors affecting technology selection	4
	2.2	Technology capabilities	5
3	**Adsorption and associated techniques**		6
	3.1	General principles	6
	3.2	Adsorption	7
	3.3	Desorption/recovery	10
	3.4	Continuous adsorption-desorption processes	13
	3.5	Adsorption capture systems	14
	3.6	Operation and installation	15
	3.7	Cost factors	17
	3.8	Summary	18
4	**Condensation and associated techniques**		19
	4.1	General principles	19
	4.2	Technical considerations	19
	4.3	Coolant/refrigerant condensation	20
	4.4	Cryogenic condensation	22
	4.5	Closed-cycle inert gas condensation	24
	4.6	Operation and installation	25
	4.7	Cost factors	27
	4.8	Summary	27

5 Absorption (scrubbing) and associated techniques 28

5.1 General principles 28

5.2 Absorption (scrubbing) 29

5.3 Desorption 31

5.4 Re-use of solvent 31

5.5 Operation and installation 32

5.6 Cost factors 33

5.7 Summary 34

6 Recent developments 35

6.1 Membrane processes 35

6.2 Plasticiser/solvent recovery system 35

6.3 Adsorbent regeneration 36

6.4 Condensation systems 36

6.5 Absorption systems 36

7 What Next? 37

Appendices

Appendix 1 Equipment suppliers and solvent recovery companies in the UK 39

Appendix 2 Bibliography 41

1 INTRODUCTION

This Good Practice Guide is intended to both promote awareness of and provide detailed information about proven technologies for the capture, recovery, and subsequent re-use of solvents from solvent-laden gas streams. The following aspects are discussed for each of the three main solvent recovery techniques - adsorption, condensation and absorption (scrubbing):

- general principles;
- applicability;
- advantages and disadvantages;
- equipment operation and installation;
- utility requirements;
- factors affecting capital and operating costs.

Volatile organic compound (VOC) emissions from industry are subject to statutory control. A first step to compliance should be a review of your solvent consumption to minimise use (see Good Practice Guide GG13, *Cost-effective Solvent Management*). If abatement equipment is required for compliance, solvent capture and re-use should be considered. Given the information in this Guide, it is hoped that the option of recovering solvent from solvent-laden gas streams will be fully considered as an economic means of VOC emissions to the atmosphere.

For advice and information on current legislation governing VOC emissions, readers are advised to contact the Environmental Helpline on 0800 585794.

1.1 TARGET PROCESSES

This Guide is intended to be of use to **any** company considering solvent capture and re-use from solvent-laden gas streams. It has particular relevance to the following manufacturing processes and industrial installations:

- printing processes;
- surface cleaning operations;
- coating processes;
- manufacture of coatings, varnishes, inks and adhesives;
- tank vents.

This Guide will be relevant for those considering new abatement equipment on existing plant, those upgrading abatement equipment, and those considering new process plant.

1.2 MAKING SOLVENT CAPTURE MORE COST-EFFECTIVE

The following considerations can significantly improve the cost-effectiveness of solvent recovery.

1.2.1 Re-use of the solvent

Key points to consider on the options for solvent re-use include:

- the number of solvents in the air stream;
- the miscibility of the solvents in water and with each other;
- the purity of solvent needed for re-use in the process;
- the possibility of re-using the solvent mixture without any further separation.

Single solvent systems are the easiest to recover for re-use. For example, a plant where the condenser is positioned immediately above the reactor vessel allows the condensed solvent mixture to return directly to the reactor.

With solvent blends of a constant composition, it is possible to recover the blend in the same proportions. This can then be re-used without further separation.

In general, capture from single solvent streams is most attractive economically. However, recovery and re-use from mixtures of up to three solvents may be viable, particularly if one is present at high concentrations and expensive to purchase new. Where mixtures need to be separated into their individual components, larger users are more likely to find the necessary separation equipment cost-effective.

There are many factors to be taken into account in assessing the economics of solvent capture and re-use. It is important that the full value of the recovered solvent, the cost of any treatment process and the cost of buying fresh solvent, are all taken into account when considering the economics of solvent recovery. In addition, if the captured solvent is sent to a solvent recovery company, and not for disposal, a credit on your total solvent consumption will be applied, and this could enable you to fall below the authorisation limit.

Ask yourself if you could improve the cost-effectiveness of solvent recovery in your company.

- How are various solvents used in the manufacturing process? Could the number of different solvents used be reduced by substituting a common solvent capable of achieving the same tasks? Fewer solvent types will make capture and re-use more viable.
- Could the recovered solvent mixture be re-used in the process itself or for other duties? Using recovered solvent as a cleaning solvent or for thinning paint, ink, etc, will offset the cost of new materials. Such duties may not consume all the recovered solvent; this, too, should be taken into account.
- Could your company use a secondary solvent recovery and recycling company? (Details of such companies are available from the Environmental Helpline on 0800 585794). These companies offer a range of services which may give a significant value to the captured solvent, so reducing your operating costs. These services include:
  - the recovery of solvents to an agreed specification for return and re-use;
  - supplying solvent blends for use by other organisations;
  - purification of a single recovered solvent to the required specification.
- Could the recovered solvent mixture be used by another company? Waste exchange organisations try to connect companies with specific wastes with other companies that may be able to use these wastes. (Details are available from the Environmental Helpline on 0800 585794). Again, this has the benefit of reducing net costs by giving a value to the recovered solvent.

1.2.2 Airflow rates

The size and cost of a solvent recovery system is usually dictated by the volumetric airflow. The suitability and efficiency of a recovery technology is highly dependent on the concentration of solvents in the air stream.

Lowering the maximum volumetric airflow reduces capital expenditure on equipment and associated operating costs such as fan power, utility requirements, etc. The resulting increase in solvent concentration also improves the efficiency of the recovery system. The safety and process constraints of the production system must, however, always be taken into account when increasing solvent concentrations.

The maximum airflow can be reduced by:

- Air recirculation.
- Improved design and optimisation of extraction and ventilation systems so that solvent vapour is removed in a smaller volume of air. Operator safety must not, however, be compromised.
- Fan control (when airflow and solvent emission rates are variable).
- Avoiding excess dilution by not mixing the air stream with other non-solvent process emissions.
- Using inert gases; this allows a reduction of airflow because VOC concentrations can be higher without flammability problems.
- Ensuring that the fan is not excessively oversized.

Significant savings are achievable with these measures. For new applications, such measures can improve the overall economics of the capture and re-use option and, for existing recovery plants, can provide a short payback on capital expenditure.

2 TECHNOLOGY SELECTION

Selecting the most appropriate technique for the recovery of solvent from a solvent-laden air stream depends on a number of factors. This Section is intended to be a quick reference guide to the criteria used to select a particular solvent recovery system. The following sections give details about each technique.

section 2

2.1 FACTORS AFFECTING TECHNOLOGY SELECTION

The following are factors that affect the selection of a solvent recovery technology.

2.1.1 Air stream

The specific factors relating to the nature of the air stream include:

- The volumetric airflow rate (minimum, maximum and average figures) and time dependency. This largely dictates the capital cost of the recovery equipment.
- The minimum, maximum and average concentration of the solvent(s) in the air stream.
- Whether the air stream contains a single solvent or a mixture of solvents.
- The presence of impurities, such as particulate matter, which may foul recovery equipment.
- The temperature and humidity of the air stream.

2.1.2 Solvent properties

The following solvent properties are important:

- boiling and freezing points;
- solubility in water and other liquids;
- whether the solvents form constant-boiling mixtures with water;
- adsorption capability;
- hazards, eg flash point, ignition temperature, upper and lower explosive limits, etc.

2.1.3 Industrial application

The particular industrial application also has a major influence on the selection of recovery equipment. Factors include:

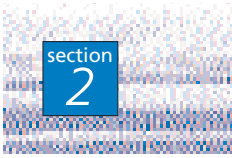
- value of recovered material;
- recovery targets;
- separation/purification steps needed to produce solvent suitable for re-use;
- availability of on-site services, eg electricity, steam, cooling water, nitrogen, and effluent treatment facilities;
- whether the industrial process is already contained or whether space considerations/process configuration would permit the installation of a closed circuit system;
- whether the recovery plant is part of a new process installation or a retrofit;
- the location and space available for siting the recovery plant.

2.2 TECHNOLOGY CAPABILITIES

The technical factors listed in Section 2.1 are important in selecting the appropriate techniques for a particular solvent recovery application. Table 1 shows the approximate range of flow rates and solvent concentrations that each technology can handle. **These are indicative figures only**.

Technology	Flow rates (m^3/hr)	Solvent concentrations (mg/m^3)
Adsorption/desorption	100 - 500 000	500 - 10 000
Adsorption capture systems	10 - 1 000	10 - 10 000
Coolant/refrigerant condensation	100 - 50 000	5 000 - 100 000
Cryogenic condensation	10 - 5 000	5 000 - 50 000
Absorption (scrubbing)	100 - 20 000	500 - 10 000

Table 1 Technology capabilities in terms of flow-rate and solvent concentration



3 ADSORPTION AND ASSOCIATED TECHNIQUES

Solvent recovery by adsorption is a proven technology, which has been used since the 1930s, and is still extensively used. It is suitable for use in applications where:

- solvents are readily adsorbed onto activated carbon or other adsorbents;
- contaminant and humidity levels in the air stream are low.

3.1 GENERAL PRINCIPLES

section 3

During adsorption, contaminant molecules from the air stream make contact with, and are retained on, the surface of a solid adsorbent where they are physically taken up at 'active sites' (see Fig 1). This attraction to the solid surface can lead to condensation of the contaminants in the micropores. As the active sites are occupied by adsorbed solvent molecules, the adsorbent becomes progressively exhausted. Contaminant breakthrough occurs once the effective working capacity of the adsorbent is reached. Desorption of the adsorbent to regenerate the active sites is then required.

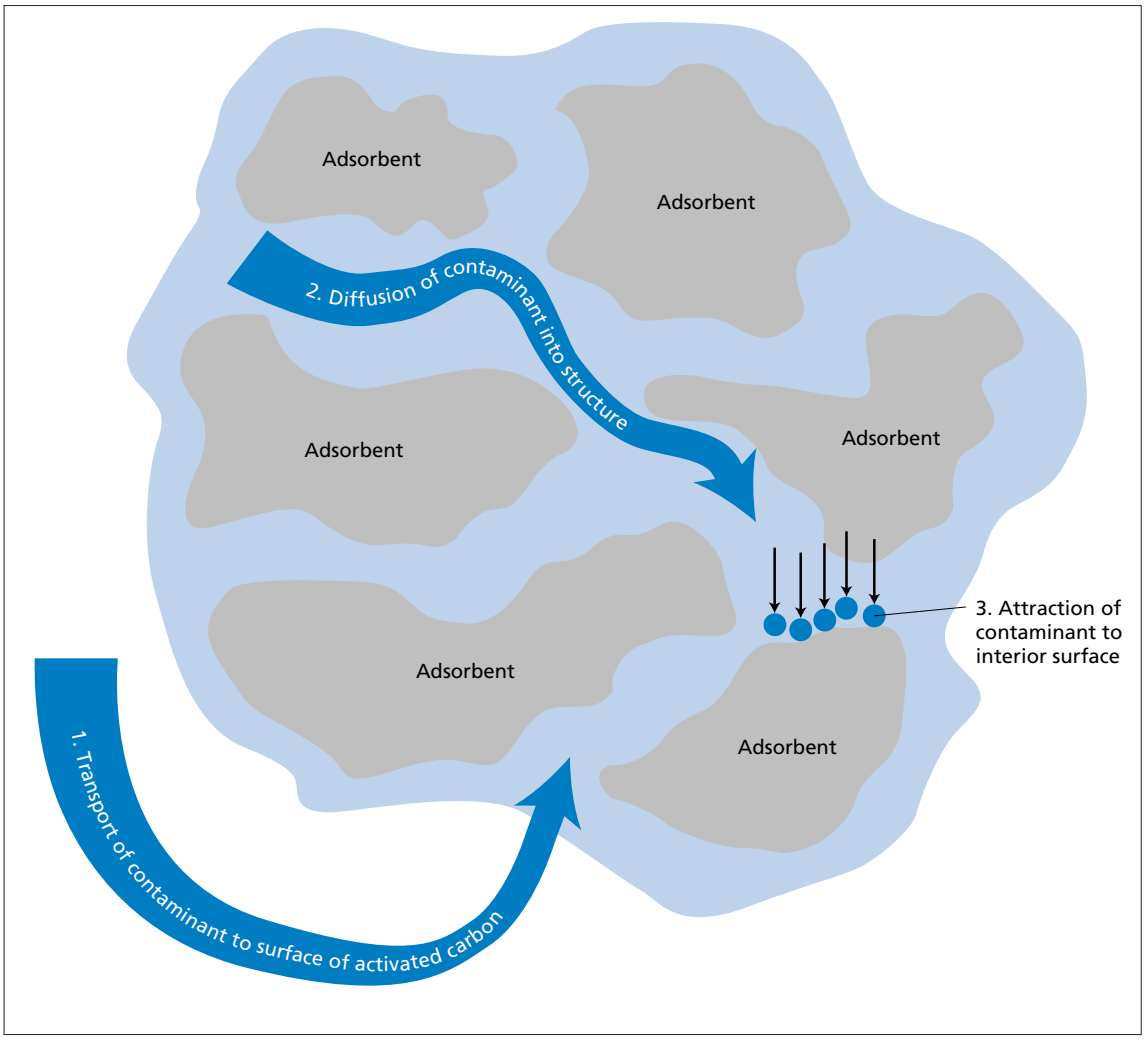


Fig 1 Principles of adsorption

Adsorption generally releases heat; desorption requires heat to purge the adsorbent of solvent.

Adsorption and desorption capacities depend on several factors, including:

- the choice and quantity of adsorbent;
- the concentration and nature of the solvent-laden gas stream;
- the process temperature, pressure and gas flow rate.

Adsorption capacity generally increases with:

- increasing molecular weight/boiling point of the adsorbed compound;
- reducing polarity;
- increasing cyclic (rather than straight chain) structure of organic solvent.

As the adsorption capacity increases, the size and cost of a unit needed to achieve a given level of adsorption decreases.

3.2 ADSORPTION

3.2.1 Adsorbents

Typical adsorbents are highly porous, granular solids with large surface-to-volume ratios. They are sized to provide low resistance to gas flow. Examples include:

- granular activated carbon (GAC);
- molecular sieve zeolites;
- macroporous polymer particles;
- silica gel;
- sodium-aluminium silicates.

GAC is the most commonly used adsorbent.

Zeolites can be manufactured with precise pore sizes, allowing selective adsorption of some compounds. As zeolites adsorb little water, they can be used at higher humidities than GAC. Zeolites are non-combustible, making them suitable for use with compounds that might represent a fire risk with GAC, eg cyclohexanone. However, zeolites cost considerably more than GAC; their use, to date, has been mainly where GAC is unsuitable.

Polymer adsorbents are particularly suited to continuous adsorption-desorption processes (see Section 3.4).

Adsorbents can hold up to 30% weight-for-weight (w/w) of solvent. Optimum systems are usually designed to achieve between 5 - 10% w/w cyclic efficiency, ie the amount of solvent adsorbed during the complete adsorption/desorption cycle.

3.2.2 Solvent compatibility

While a wide range of solvents can be removed by adsorption (see Fig 2), not all are recoverable by desorption. Desorption of high boiling point solvents (higher than 200°C) requires more energy. Phenols and nitriles are particularly difficult to desorb. Cyclohexanone is prone to polymerise when adsorbed, significantly reducing adsorbent life and creating a potential flammability hazard.

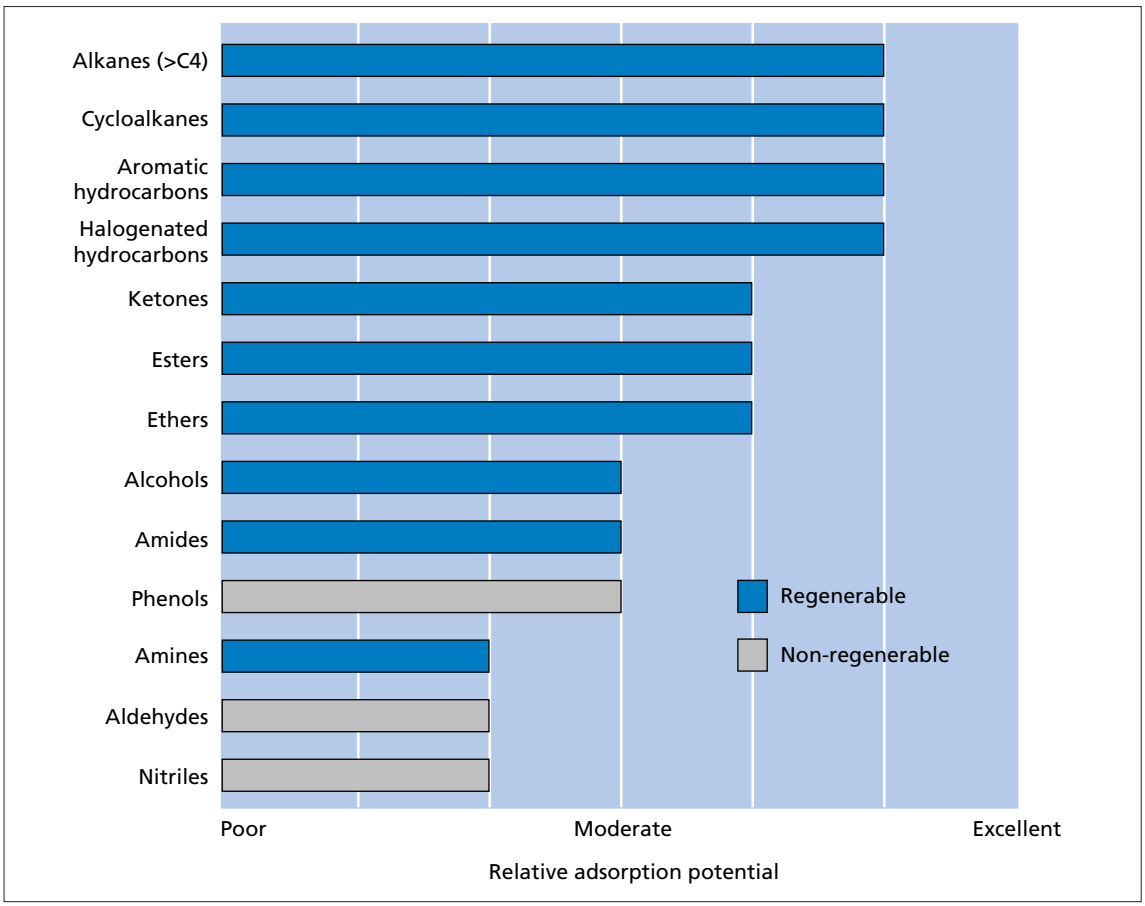


Fig 2 Adsorption potential of granular activated carbon (GAC) with various organic solvents

Hydrolysis of normally stable solvents can occur when the solvent is adsorbed on a GAC surface. In the case of chlorinated solvents, the resulting hydrochloric acid can lead to corrosion problems. Ketones and aldehydes have a tendency to oxidise in the presence of GAC, producing carboxylic acids and liberating substantial amounts of heat. Failure to control these reactions and to cool the GAC adequately can result in a chain reaction leading ultimately to oxidation of the GAC matrix and auto-ignition of the GAC. The latter occurs at around 370°C.

Low volatility solvents are more readily adsorbed than solvents with a high volatility. They therefore will tend to displace the more volatile ones as the bed becomes more saturated. This problem can be overcome by careful design, eg:

- Providing sufficient adsorption capacity for the mixture.
- Using adsorption beds in series. Use of a 'guard' bed ensures that if solvent is displaced from the first bed, it is adsorbed in the second.

3.2.3 Adsorption systems

General system design considerations include:

- ample contact time between the gas stream and adsorbent bed to achieve the required removal efficiency;
- adequate adsorbent capacity to ensure a reasonable adsorption on-stream time;
- possible pre-treatment to avoid pore blinding;
- uniform airflow distribution to ensure complete utilisation of the adsorbent.

Other important parameters are:

- The temperature of the air stream (cooler air streams are more efficient).
- The moisture content of the air stream. The performance of adsorbents (particularly GAC) is sensitive to the moisture content of the air. Once the relative humidity of the stream exceeds 60%, the adsorption capacity is significantly reduced owing to the microporous structure of the adsorbent filling with water.

Potentially necessary pre-treatments include:

- pre-cooling the air to improve adsorption efficiency;
- using filters or mist eliminators to remove solids or liquids (droplets or aerosols) which can cause plugging of the adsorbent;
- using a sacrificial layer of adsorbent to remove components with boiling points greater than 150 - 200°C that would be collected but not removed (due to the excess energy requirement) during bed regeneration.

3.2.4 Fixed bed process

This process variant involves the alternate batch-wise adsorption and desorption of the solvent in a fixed bed of adsorbent. More than one adsorber is required for continuous operation. Twin beds are typical, with one bed adsorbing the solvent while the other is being regenerated. Third or fourth beds may be required if:

- the time required for the adsorption phase differs from that for the desorption phase;
- stand-by capacity is required.

A typical twin-bed adsorption process is shown in Fig 3.

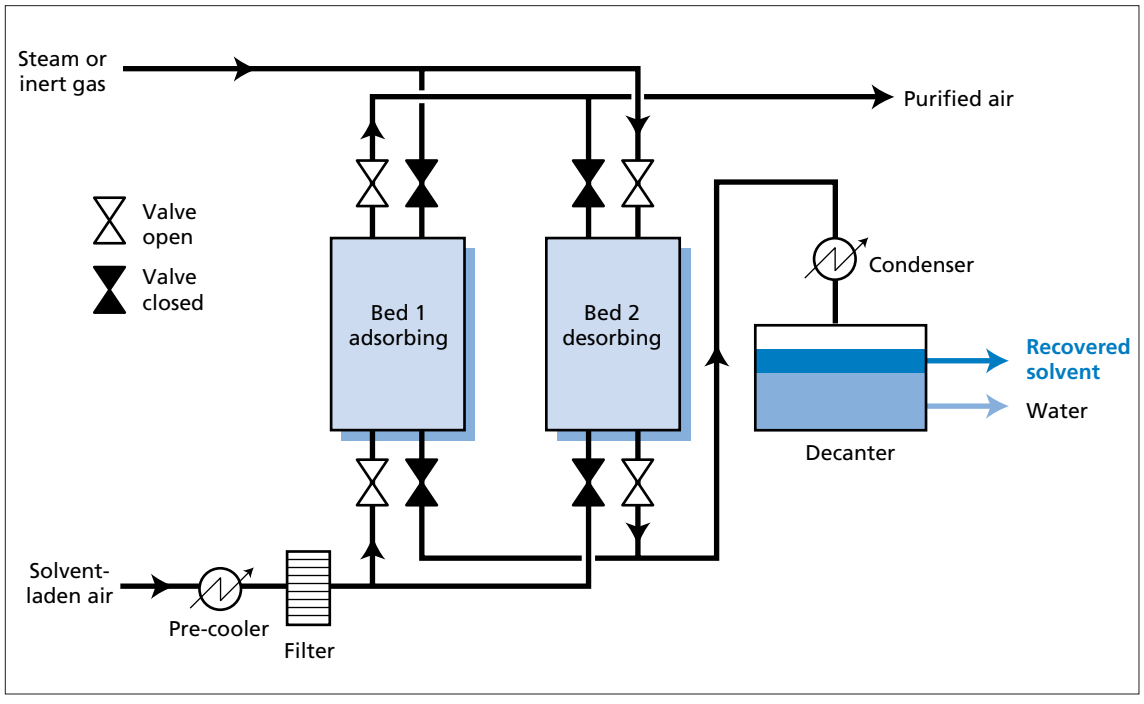


Fig 3 Typical twin-bed adsorption process

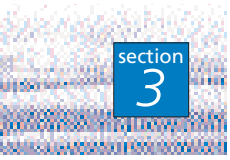
In Fig 3, solvent-laden air flows upwards through the adsorbing bed, venting to atmosphere from the top or side of the adsorber shell. When breakthrough is achieved, the adsorber is switched to desorption, steam or inert gas passing downwards through the desorbing bed.

Adsorption cycles are controlled by one of the following:

- a gas analyser using a set solvent breakthrough concentration to switch adsorbers;
- a calculated fixed time cycle.

3.2.5 Activated carbon mat filter

An alternative to GAC is the activated carbon mat filter. The filter comprises a circular filter element consisting of a non-woven web of fine activated carbon filaments. Solvent vapour is adsorbed within the very fine micropores of the carbon. The different physical nature of the carbon allows a higher specific surface area to packed volume ratio to be achieved, ie there are more active sites per unit volume. Higher solvent loadings can thus be achieved per unit mass of carbon cloth compared to GAC. This allows for a more economical use of steam and a reduction in the total volume of effluent produced by the plant.



Such systems can be used to recover a wide range of solvents, including polar compounds and ketones. Hot spot formation is avoided due to the short cycle times. However, activated carbon mat filters are typically 100 times more expensive than GAC.

3.3 DESORPTION/RECOVERY

Desorption - the reverse of adsorption - can be carried out by:

- steam desorption;
- inert gas desorption;
- vacuum desorption.

These three methods, which are shown in simplified flow charts in Fig 4, are described in more detail below.

For safety reasons, hot air desorption is not, in general, a preferred technique for solvent recovery. However, it can be used when solvent concentrations are low (hundreds of parts per million (ppm)), for example in adsorption/desorption processes which are used to concentrate emission streams prior to further treatment. More recent developments include continuous adsorption-desorption processes (see Section 3.4).

3.3.1 Steam desorption

This relatively cheap and often simple method (see Fig 4) is the most widely used regeneration technique. It uses saturated steam at temperatures of 100 - 110°C to heat the adsorbent by steam condensation. Initially, all the steam is used to heat both adsorbent and adsorber to the steam saturation temperature at the existing vessel pressure. Desorption of the adsorbed solvents does not start until the adsorbent bed has warmed up.

During the desorption process, some steam condenses on the adsorbent to deliver the energy needed for solvent desorption. Most of the steam, however, flows through the adsorbent removing the desorbed solvent. Further reduction of the residual solvent load is obtained by the flushing effect of the steam and the declining solvent partial pressure.

Typical results, for an exit concentration of 20 ppm, of a solvent adsorption/desorption system using GAC are shown in Table 2. Exit concentrations can be as low as 5 ppm.

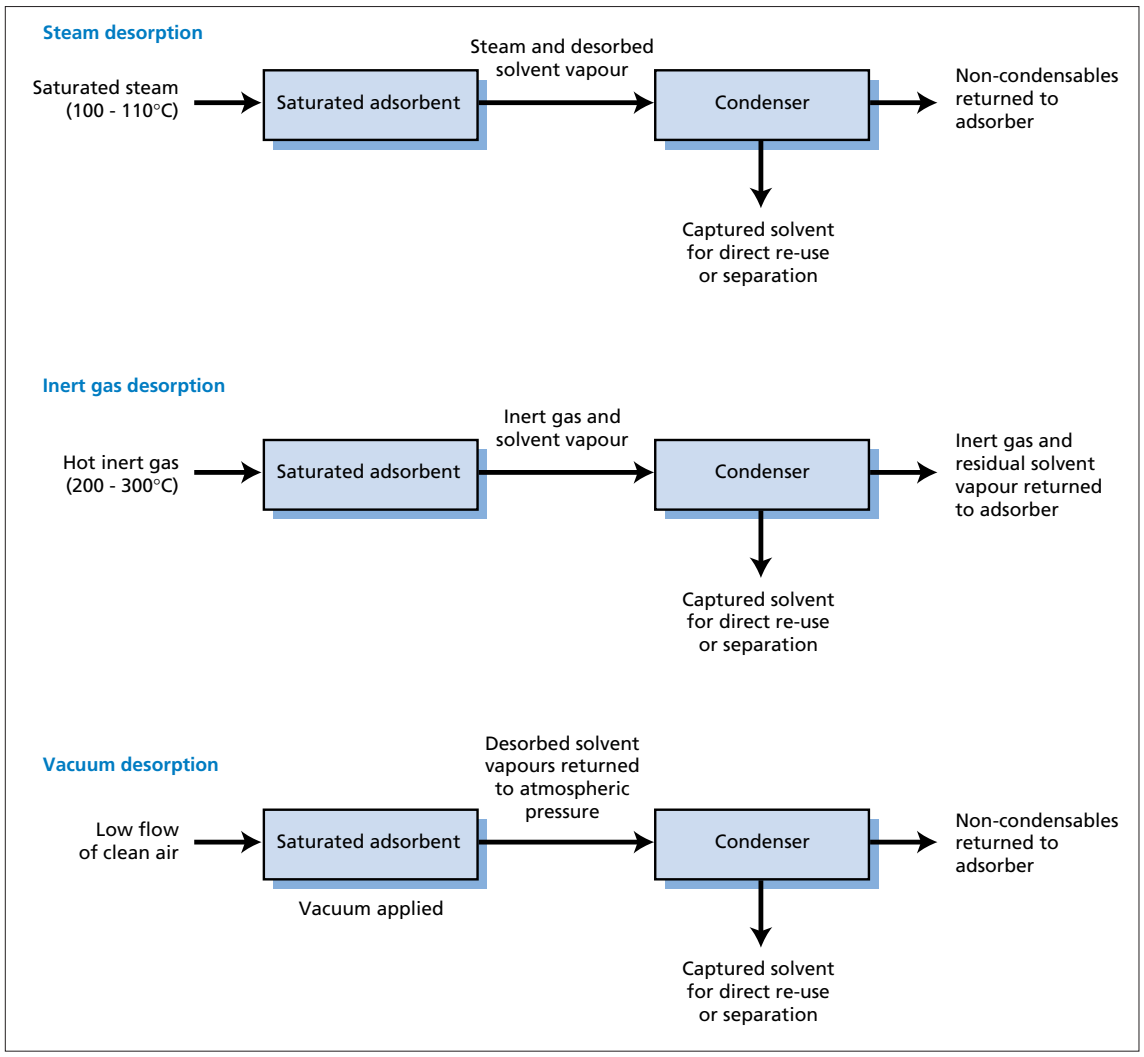


Fig 4 Desorption/recovery methods

Solvent	Inlet concentration (ppm)	Optimum solvent content of bed (% w/w)	Steam required to desorb 1 kg of solvent (kg)
Methylene dichloride	10 000	17	1.4
Acetone	10 000	21	1.4
Tetrahydrofuran	5 000	9	2.3
n-Hexane	5 000	8	3.5
Ethyl acetate	5 000	13	2.1
Trichloroethylene	5 000	20	1.8
n-Heptane	5 000	6	4.3
Toluene	4 000	9	3.5
Methyl isobutyl ketone	2 000	9	3.5

Table 2 Typical results of solvent adsorption using GAC

Total desorption is often neither technically nor economically feasible. A residual quantity of solvent - the 'heel' - remains on the bed, reducing the number of active sites available for adsorption. The increase in this residual heel with time (years) causes the absorbent to lose its ability to be regenerated in situ. External re-activation or replacement is then required.

The desorbed vapours, which are condensed in conventional water-cooled or air-cooled heat exchangers, are collected for re-use or further purification. Uncondensed vapours from the desorber are recycled into the incoming air stream. Following steam desorption, the bed should be dried with air and allowed to cool before adsorption recommences.

Steam is the preferred regenerating agent for solvents that are immiscible with water; the solvent phase and the steam condensate can be readily separated in a decanter. Both the concentration of water in the solvent phase and the concentration of solvent in the aqueous phase depend on the degree of immiscibility and the temperature. Provided a small amount of water in the solvent phase does not affect the manufacturing process, the solvent may be re-used direct from the decanter. The aqueous phase can be treated as necessary.

Distillation is normally used to separate water-soluble solvents - eg alcohols or esters - and multi-solvent mixtures that cannot be separated by decantation. The large heat input needed for distillation may result in high operating costs. Distillation can be performed:

- on-site;
- off-site by a solvent recovery company (contact the Environmental Helpline on 0800 585794 for more information).

Water-soluble solvents that form constant-boiling mixtures are not readily separated. They require distillation, followed by further purification, to obtain re-usable solvents.

3.3.2 Inert gas desorption

Nitrogen is the inert gas typically used for this method (see Fig 4), which is particularly suitable for use with water-soluble solvents.

Oxygen levels in the desorption loop are first reduced by purging with dry nitrogen. Hot inert gas at 200 - 300°C is then circulated. The hot inert gas preferentially desorbs the moisture captured from the air by the adsorbent. This water is subsequently removed by drying the gas with a molecular sieve. Once the water is desorbed, hot, dry inert gas desorbs the solvent. The desorbed solvent is condensed from the carrier gas, ready for direct re-use.

Heat removed during condensation can be transferred back to the gas heater by a heat pump, while the hot gas can be recycled through the adsorber. Once the bed is fully desorbed, gas heating should cease and the circulating gas allowed to cool the bed. The heat removed from the bed can be used to regenerate the molecular sieves.

If a low moisture content is acceptable in the recovered solvent, then it is not necessary to dry the carrier gas using molecular sieves. The solvent can then be recycled direct from the condenser. Further separation is required if:

- any hydrolysis of the solvent occurs;
- the water content is significant;
- a mixture of solvents is recovered.

This separation is usually achieved by distillation.

The main advantage of inert gas desorption is that the recovered solvent has a low water content and may be fit for direct re-use.

3.3.3 Vacuum desorption

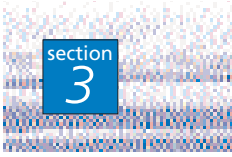
In this method (see Fig 4), the reduced pressure causes the adsorbed solvent to vaporise from the adsorbent pores. One example is known as the ‘pressure swing adsorber’.

In systems using vacuum desorption, the adsorbent beds are designed to retain the heat released during the adsorption step for use in a subsequent regeneration step. During regeneration, a vacuum is created around the bed. This causes the adsorbed solvent to re-vaporise and desorb from the adsorbent. The desorption process is bolstered by the heat of adsorption retained in the bed. The solvents desorbed from the bed are removed by back-purging with a small portion of the clean vent stream.

The concentrated desorbed solvent vapours are returned to atmospheric pressure and condensed; non-condensables are returned to the on-line adsorber.

Vacuum regeneration works best when recovering high volatility solvents from high-capacity adsorbents. However, the technique usually has higher equipment costs than other methods.

3.4 CONTINUOUS ADSORPTION-DESORPTION PROCESSES



Continuous systems eliminate the need for duplicate equipment as the adsorption and desorption stages are carried out in the same unit.

The solvent-laden air stream, which is introduced at the base of the adsorption section, flows upwards through perforated trays arranged one above another. The solvent is adsorbed and the treated air discharged to atmosphere.

Regenerated adsorbent is fed continuously by a conveyor to the top adsorber tray, from where it flows downwards from tray to tray. The saturated adsorbent on the lowest tray passes into the regeneration section via a nitrogen gas lock separating the two sections. The adsorbent then travels downwards through the desorber tubes. The adsorbent is indirectly heated to desorption temperature and desorbed with an inert gas, eg nitrogen. The regenerated adsorbent is cooled and returned pneumatically to the top adsorber tray, while the hot inert gas stream is cooled in an external condenser to remove solvent. By adjusting the rate at which the regenerated adsorbent is returned to the adsorber, its retention time in the adsorber can be controlled.

The recovered solvent, which is virtually water-free, can be directly re-used. Steam can be used instead of an inert gas when water-insoluble solvents are being recovered. The condensed steam/solvent separation is performed in a decanter.

The main problems with continuous systems are due to their greater complexity compared to traditional systems. Continuous adsorption-desorption systems require a large quantity of adsorbent, with allowance made for any attrition and breakdown of the adsorbent.

3.4.1 Polymer adsorbent systems

One variation is to use a polymer adsorbent instead of activated carbon (see Fig 5). In this system heated air is used in the desorption process. The polymer adsorbent, which is intended for air streams with low solvent concentrations, has the following advantages:

- durability;
- unaffected by high humidity;
- does not catalyse the degradation of unstable solvents;
- regenerated under very mild conditions;
- high purity solvents recovered.

Polymer adsorbents are, however, ten times more expensive than GAC. They are suitable for use with most common solvents, except for very polar compounds and those with low boiling points, eg methanol and methyl chloride.

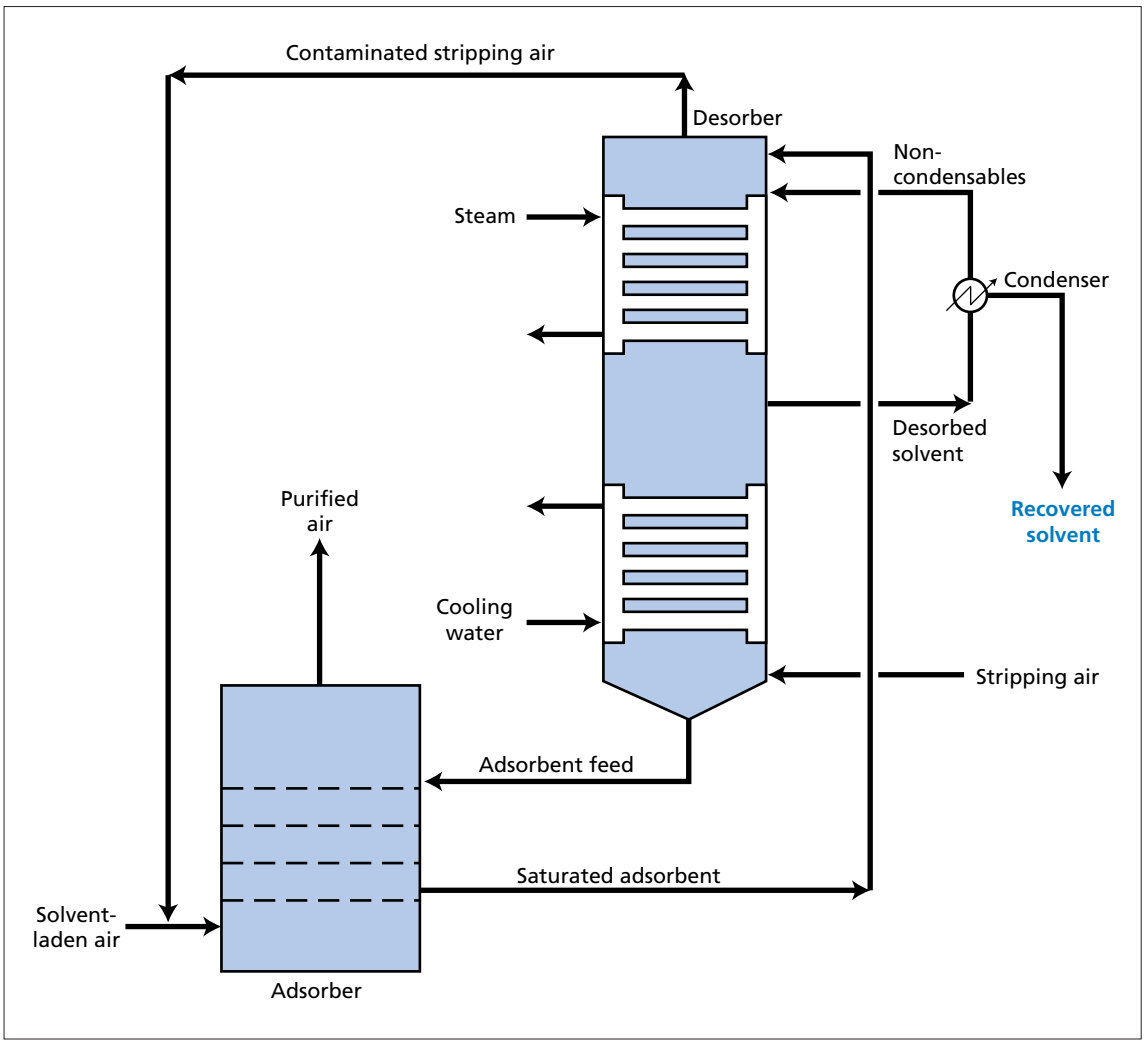


Fig 5 Continuous adsorption-desorption with a polymer adsorbent

3.4.2 Carbon wheel concentrator

The system known as the 'carbon wheel concentrator' consists of a corrugated wheel made of a ceramic honeycomb structure coated with powdered activated carbon. The wheel, which rotates continuously at slow speed (1 - 2 rev/hr), is divided into three basic zones:

- adsorption;
- desorption;
- cooling.

In the first zone, solvent is removed by adsorption as the solvent-laden air stream passes through the honeycomb structure. A small volume of air or another gas, heated to 110 - 130°C, passes through the desorption section in the opposite direction to the process stream. The solvent is continuously desorbed from that part of the wheel and the adsorbent regenerated. The regenerated zone is then cooled to maintain the desired temperature.

3.5 ADSORPTION CAPTURE SYSTEMS

These units are based on standard adsorption principles. However, in this case, the adsorbent is not regenerated on-site. Instead it is taken off-site and either disposed of or regenerated. These units are usually off-the-shelf systems designed for applications with low/intermittent solvent loadings. Typical applications include:

- storage tank venting;
- treatment of outlet air from stripping towers (depending on humidity levels).

Such adsorption systems typically consist of GAC in a deep, but compact, canister. Efficient contact between the gas and GAC is achieved by using a perforated distribution plate. This, together with an extended contact time, results in high efficiency adsorption but with a low pressure drop.

Both installation and operation are straightforward owing to the system's simple design. Operation merely involves routine monitoring of outlet concentrations. Once breakthrough occurs, continuity of operation is maintained by replacing either the whole unit or just the GAC. These operations are normally carried out by the supplier.

Table 3 shows the typical adsorption efficiencies for six common solvents using GAC as the adsorbent.

Solvent	Adsorption efficiency (%)*		
	1 g/m^3 feed	10 g/m^3 feed	100 g/m^3 feed
Dichloromethane	11	31	45
Styrene	34	38	41
Tetrachloroethylene	50	61	65
Toluene	32	34	37
1,1,1-Trichloroethylene	33	47	55
1,1,2-Trichlorotrifluoroethane	11	29	45

** Assuming a carbon density of 0.53 g/cm^3.*

Table 3 Effect of varying feed concentrations on mass of solvent adsorbed

The operating costs of adsorption capture units consist mainly of the cost of supplying and regenerating the GAC. The frequency of re-supply depends on the capacity of the unit and the solvent loading, while the airflow dictates the size of the unit. Such systems are likely to be most cost-effective for low concentration streams (up to a few hundred ppm) or infrequent emissions.

3.6 OPERATION AND INSTALLATION

3.6.1 Concentrations, flows and efficiencies

The concentration of the air stream is dependent upon the lower explosive limit.

Adsorption systems can be designed to handle airflows up to 500 000 m^3/hr while achieving removal efficiencies of greater than 99%. GAC plants are typically designed with a linear gas velocity of 0.05 - 0.4 m/sec to give a residence time of 0.5 - 4 seconds in beds 50 - 150 cm deep.

An adsorption system designed for a particular maximum airflow and solvent concentration should be able to cope with reasonably wide variations in these two parameters. However, too great a fluctuation in solvent concentration over a period of time may affect the bed equilibria leading to a risk of spontaneous desorption. If this is a potential problem, a guard bed can be used to capture any such desorbed solvent.

Activated carbon mat filters can typically achieve 97 - 98% efficiency.

Continuous adsorbent systems, which are suitable for solvent concentrations in the range 0.1 - 2 g/m^3, can achieve 98% efficiency with airflows from 1 000 - 700 000 m^3/hr.

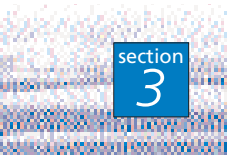
3.6.2 Utilities and consumables

The main utilities required for adsorption systems are:

- steam or hot oil to heat the desorbing inert gas, depending on the desorption method;
- cooling water for condensing desorbed solvent;
- electricity to power fans, pumps, etc.

As a general rule, steam regeneration for GAC requires a steam to solvent ratio between 1.5:1 and 6:1 (w/w). If desorption is carried out using an inert gas, eg nitrogen, operating experience suggests that 35 m^3 of nitrogen is needed to desorb one tonne of solvent.

Steam requirements for an activated carbon mat filter can be expected to be at least twice that of GAC or polymer adsorption systems, ie a steam to solvent ratio of between 6:1 and 12:1 (w/w).



It is not possible to predict the life of the adsorbent. This depends on the level of adsorption inhibitors in the system, eg:

- particulates;
- moisture (humidity);
- high molecular weight solvents.

Although the adsorbent can be re-activated by thermal treatment and re-used, re-activated GAC is not generally as efficient as new GAC.

3.6.3 Control and operation

Adsorption systems are typically controlled by a programmable logic controller, with new systems based on breakthrough sequencing of the beds. When the emission level from an operating bed reaches a pre-set level (ie at adsorbent breakthrough), the beds are switched over and regeneration starts. This is energy efficient as desorption is always performed on a saturated bed.

Beds are also switched by a back-up timer to avoid oversaturation. Given a constant airflow and solvent concentration, an automatic timer system is more cost-effective. Such systems are normally designed with approximately equal adsorption and desorption times.

Subject to consideration of flammable hazards, control systems can be designed to allow a solvent concentration of up to 40% of the lower explosive limit. This minimises both fan power and steam consumption (at higher inlet solvent concentrations, the capacity of the adsorbent is higher).

If flow rates and solvent loadings are variable, power requirements can be optimised by fitting control dampers or variable speed drives for the fan. For example, in a printing process drying cycle where 40 - 50% of the solvent is emitted in the first hour, the controls are set to maintain a constant solvent concentration with a variable airflow. The flow rate is therefore reduced as the solvent concentration decreases.

Where GAC is used to adsorb ketones, careful start-up and shut-down procedures must be followed to minimise the risk of bed fires. If insufficient desorption and cooling has been carried out at shut-down, ketone oxidation can result in pockets of heat ('hot spots') within the bed. A chain reaction followed by a bed fire may result.

With standard control systems that include automatic alarms and shut-down capabilities, staff requirements are minimal, merely consisting of periodic checks of gauges, etc, once or twice a day. Larger systems may need staff attention for about an hour a day.

3.6.4 Maintenance

Maintenance is minimal as there are few moving parts. It is typically concerned with pumps, fans, etc, plus occasional adsorbent sampling to check its remaining life.

3.6.5 Ease of retrofitting

Provided sufficient space is available, retrofitting an adsorption system to an existing manufacturing process is generally straightforward. The space requirement of a continuous adsorption/desorption unit is approximately 25% that of a two-bed adsorber system.

3.7 COST FACTORS

The following factors influence the cost of an adsorption recovery system.

3.7.1 Capital cost

Emission flow rate. This influences the overall size of the system.

Solvent type. This dictates the choice of adsorbent. For example, GAC is considerably cheaper than activated carbon mat filters or polymeric adsorbents.

Solvent adsorption efficiency and solvent concentration. Both influence the quantity of adsorbent required.

Solvent mixtures. Multi-component solvent streams are likely to require more complex separation techniques.

Solvent solubility. The more soluble the solvent is in water, or other components present in the air stream, the more complex the separation technique likely to be required.

3.7.2 Operating costs

Emission flow rate. This dictates utility requirements.

Solvent loading. This influences both the use of utilities in the adsorption/desorption cycle, eg steam, and the rate of adsorbent degradation.

Presence of impurities. Particulates and high boiling point solvents will reduce the life of the adsorbent.

Ease of solvent desorption. High boiling point solvents require higher temperatures for desorption, but condense more readily.

Solvent mixtures. More energy-intensive separation techniques are likely to be required.

Solvent solubility. The more soluble the solvent is in water, or other components present in the stream, the more energy-intensive the separation technique likely to be required.

3.8 SUMMARY

- Adsorption-based techniques are extensively used for solvent recovery.
- GAC is the most common adsorbent. Others include zeolites and polymer particles.
- Adsorption capacity increases with increasing molecular weight and boiling point of solvent, but decreases with increasing polarity. Activated carbon adsorption is impaired when the gas stream has a humidity above 60%.
- Desorption can be carried out by using steam, a hot inert gas (eg nitrogen) or under vacuum (reduced pressure). Steam desorption of water-soluble solvents may necessitate more complex separation techniques for solvent recovery.
- Adsorption/desorption systems can be:
  - fixed beds operated in alternate modes;
  - systems where the adsorbent is continuously conveyed between the adsorber and desorber.
- Contaminants such as particulates and high boiling point solvents significantly reduce adsorbent life.
- Adsorption capture systems can represent a highly practical means of eliminating emissions from intermittent sources.
- Changing from air to inert gas allows the airflow rate to be decreased and solvent concentrations increased without compromising safety. Changing the airflow in this way will reduce operating costs, thus making solvent capture and re-use more cost-effective.

4 CONDENSATION AND ASSOCIATED TECHNIQUES

Condensation systems have traditionally been considered useful preliminary recovery units to reduce a high solvent loading prior to further recovery, eg using an activated carbon adsorption bed. While this remains a feasible arrangement, developments in both heat exchanger systems and use of low temperature fluids mean that condensation systems are now a viable stand-alone option for solvent recovery.

While conventional condensation techniques are most suitable for solvents with a reasonably high vapour pressure, cryogenic condensation is able to cope with all solvents irrespective of their vapour pressures.

4.1 GENERAL PRINCIPLES

Solvent vapours only remain in the gas phase up to a maximum concentration determined by the solvent vapour pressure at the temperature concerned. The maximum amount of solvent that can be held in a unit volume of air at equilibrium under stationary conditions is referred to as the **saturation concentration.** This concentration can be calculated from the solvent's vapour pressure and molecular weight. The temperature at which this saturation concentration is reached is known as the **dew point.** If the temperature falls below the dew point, some of the solvent must revert to the liquid phase until the saturation concentration (ie equilibrium) in the gas phase is reached at the lower temperature. If the vapour phase consists of a solvent mixture, allowance must be made for the actual phase equilibria of the individual components. No simple relationship exists for the saturation concentration of the system under such conditions.

This physical relationship forms the basis for solvent recovery by condensation.

In practice, there are technical and economic limits to the application of condensation to solvent recovery. If the concentration of the solvent vapour is very low, the temperature may need to be lowered significantly before condensation commences. This may require temperatures that are difficult to attain. Problems with freezing may occur, particularly when solvent blends are condensed, due to the differential freezing points within a solvent mixture. Frozen solvent on the heat transfer surface impedes heat transfer and may block the tubes, thus decreasing the condensation rate. Furthermore, water vapour below its freezing point forms an amorphous deposit on the surfaces of heat exchangers, impeding heat transfer.

Minimising the non-condensable gas content of the air stream increases the practicality and economic attractiveness of condensation. Vapour streams which are some way away from their dew points generally cost more to condense than those fairly close to their dew points.

4.2 TECHNICAL CONSIDERATIONS

Although condensation systems can recover almost any solvent, the feasibility of condensation as a recovery technique depends on the condensing temperature required. Generally, the lower the boiling point of the solvent, the more difficult it is to recover. Cryogenic condensation is an option if condensing temperatures lower than -30°C are required.

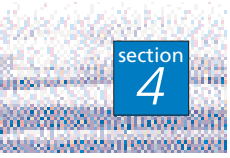
Solvent mixture recovery by condensation is simple, being limited only by the condensation temperature. However, the condensate is also a mixture, and separation into individual solvents may subsequently be required. While it may be possible to separate mixtures by recovering condensate

at progressively lower temperatures (the least volatile condenses first and the most volatile last), some cross-contamination will occur. Unless the solvents are immiscible and can be separated by decantation, further separation stages, eg distillation may be required. 'Dry' solvent can be obtained by removing water using pre-coolers and molecular sieves.

Condensers are frequently used to recover emissions for direct return to their source, eg from vents in solvent storage tanks or from process vents on reactors or columns. No further purification is required and the treated air stream is discharged to atmosphere. If water is present and has to be removed, or if a solvent mixture is recovered that cannot be directly re-used, then further separation of the condensate, eg by distillation, would be required to obtain useful solvent.

Coolant/refrigerant condensation is a proven technology with thousands of applications worldwide. Cryogenic systems are less widespread, with fewer than 100 end-of-pipe systems worldwide. Although there are about 100 closed-cycle inert gas condensers installed worldwide, there are at present only a few installations in the UK.

4.3 COOLANT/REFRIGERANT CONDENSATION



4.3.1 Conventional shell-and-tube heat exchangers

Condensation can be carried out by either direct or indirect cooling. With direct condensation, however, solvent contact with the cooling liquid necessitates an additional separation stage. Indirect condensation is therefore preferred.

In indirect condensation, the air stream typically passes around the internally-cooled tubes of a shell-and-tube heat exchanger. The solvent vapours condense as a film on the cold, shell side of the tubes. The heat exchanger is positioned either vertically or slightly sloping, thus allowing the condensate to drain into a collection tank or be directly re-used.

Recovery systems range from simple, single condensers to more complex, multi-condenser systems designed to maximise energy and solvent recovery.

Low volatility solvents can be effectively recovered using water-cooled or air-cooled systems. However, lower temperatures are generally required to recover significant quantities of solvent. Chilled water or refrigerants such as glycol and liquid methanol may be required to achieve such temperatures. For more volatile solvents, two-stage condensation using water-cooling in the first stage and refrigeration in the second stage may be necessary.

In a two-stage system (see Fig 6), the solvent-laden air stream first enters a recuperator which uses the cold purified air stream leaving the main condenser as the chilling agent. The air stream is then cooled further in a pre-cooler, eg using chilled water or the cold purified stream, before entering the main refrigerated condenser.

The successive cooling procedure:

- minimises the refrigerant requirement;
- condenses most of the high freezing point components, eg water, before freezing occurs;
- minimises fog formation (see Section 4.3.2).

The size and arrangement of the various condensers should be chosen to optimise thermal efficiency and minimise equipment cost.

An alternative involves partial condensation at a slightly higher temperature followed by removal of the remaining solvent from the air stream by another technique, eg by adsorption.

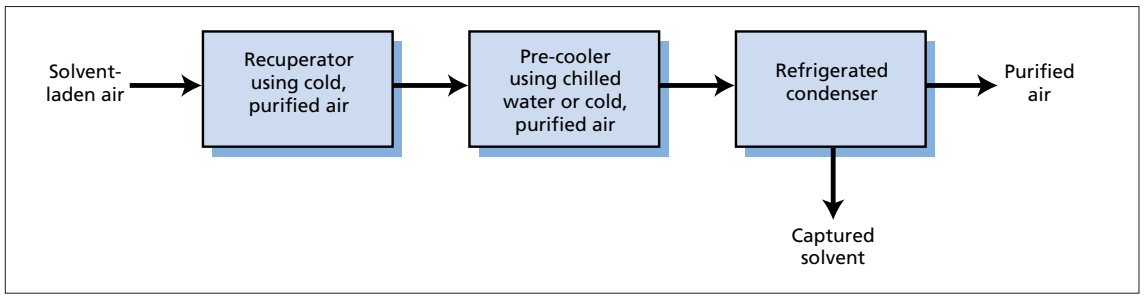


Fig 6 Typical two-stage condensation system

4.3.2 Fog formation

When the rate of heat transfer exceeds the rate of mass transfer, the bulk of the gas rapidly cools below the dew point of the condensable vapour. Droplets have insufficient time to migrate to a cold surface and therefore nucleate and condense in the bulk gas stream. This can result in solvent emission to the atmosphere as droplets. Fog formation can be minimised by:

- using a series of condensers;
- using a demister;
- reducing the gas velocity through the condenser so that the droplets fall out of the gas stream rather than accompany the gas stream out of the unit.

4.3.3 Spiral heat exchangers

An alternative to the standard shell-and-tube condenser is the spiral heat exchanger. The unit basically consists of two long strips of plate wrapped to form a pair of concentric spiral passages. The cooling medium enters through a peripheral nozzle, spirals to the centre and exits, via a pipe, to a nozzle on the periphery. Process vapours enter through the bottom of the condenser and flow upwards in cross-flow. Fouling tendencies are minimised by the single passage for each fluid. The long pass on each side facilitates close temperature approaches and temperature crosses.

Although spiral heat exchangers may be more expensive than shell-and-tube exchangers, they have the following advantages:

- even flow distribution;
- high turbulence;
- self-cleaning;
- low pressure drop;
- small space requirements, eg a spiral element of diameter 1 m and length 1.5 m has an effective surface area of 100 m^2.

Other types of heat exchanger can give some of the advantages of the spiral heat exchanger.

4.3.4 Condensation filtration recovery systems

This specialised condensation system uses surface condensation and microfiltration to recover solvents. It is particularly suited to the heatset web offset printing sector. A typical system consists of three main elements:

- a condensation unit;
- a moving bed filter;
- a mist eliminator.

In a typical system (see Fig 7), an air stream at 160 - 180°C enters the condensation unit where it is cooled to 40°C using ambient air. The high boiling point solvents condense, forming a liquid condensate and a mist of liquid solvent droplets. The condensate passes to a liquid solvent filtration

unit - where any solid contaminants are removed - and into a solvent/water separator. Approximately 60% of the liquid component is removed in the condensation stage. The recovered solvent is passed to the main solvent storage tank for re-use.

The air stream containing the mist of solvent droplets then enters the moving bed filtration unit where any solids and solvent droplets are removed. The solvent droplets are recovered via the liquid solvent filtration unit and the solvent/water separator. A differential static pressure controller automatically advances the moving bed filter on a new section of filter, thus maintaining optimum efficiency.

Finally, the air stream passes into the mist eliminator where any remaining aerosol droplets are collected for recovery in the liquid solvent treatment system. The treated air is discharged to the atmosphere.

Combined condensation and filtration systems are ideally suited for the recovery of high boiling point solvents.

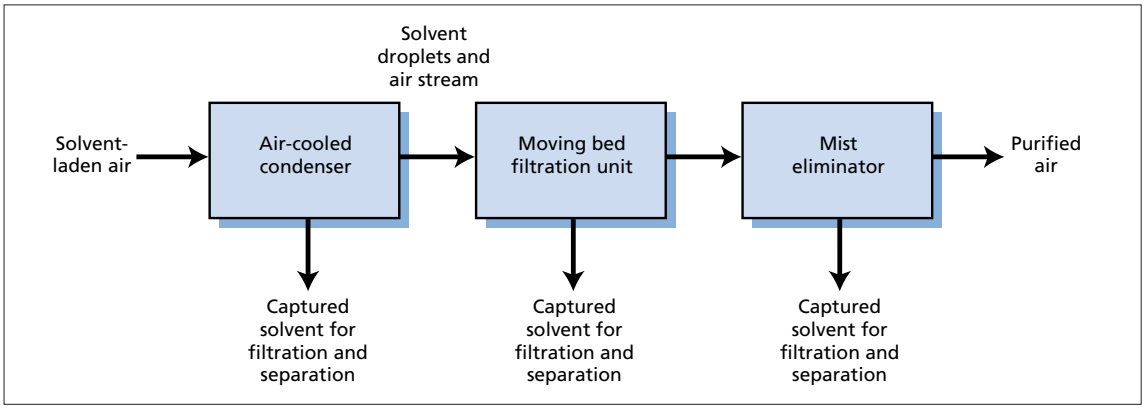


Fig 7 Typical condensation filtration recovery system

4.4 CRYOGENIC CONDENSATION

Condensation at extremely low temperatures can be carried out using cryogenic nitrogen. Nitrogen has the following properties that make it ideal for use as a low temperature refrigerant:

- low boiling point (-196°C);
- non-toxic;
- non-corrosive;
- non-flammable.

Many industrial processes use nitrogen as an inert blanketing gas to minimise unwanted reactions, prevent fires and reduce product degradation by exposure to air or moisture. The nitrogen gas is produced by vaporising delivered liquid nitrogen in a heat exchanger. The use of nitrogen to provide an inert atmosphere usually means that cooling capacity is 'freely' available and no further liquid nitrogen is required for cryogenic condensation.

Cryogenic condensation can take two forms. In the first, the 'cold' released when liquid nitrogen is vaporised is used as a coolant, with the solvent vapour condensing on the cold surface of the condenser. In this case, the maximum solvent concentration of the air stream is set at 25% of lower explosive limit. Alternatively, the cryogenic condensation plant is run under an inert atmosphere. This allows the air stream to contain higher concentrations of solvent. The latter system has the following advantages:

- lower capital and operating costs;
- reduced plant size.

Fig 8 shows a simplified plant design where liquid nitrogen is vaporised in the recovery unit. The vaporised nitrogen gas is used to blanket several processes before returning to the recovery unit carrying solvent vapours from the processes. The solvent is condensed and separated by the recovery unit, while the purified nitrogen gas is vented to the atmosphere.

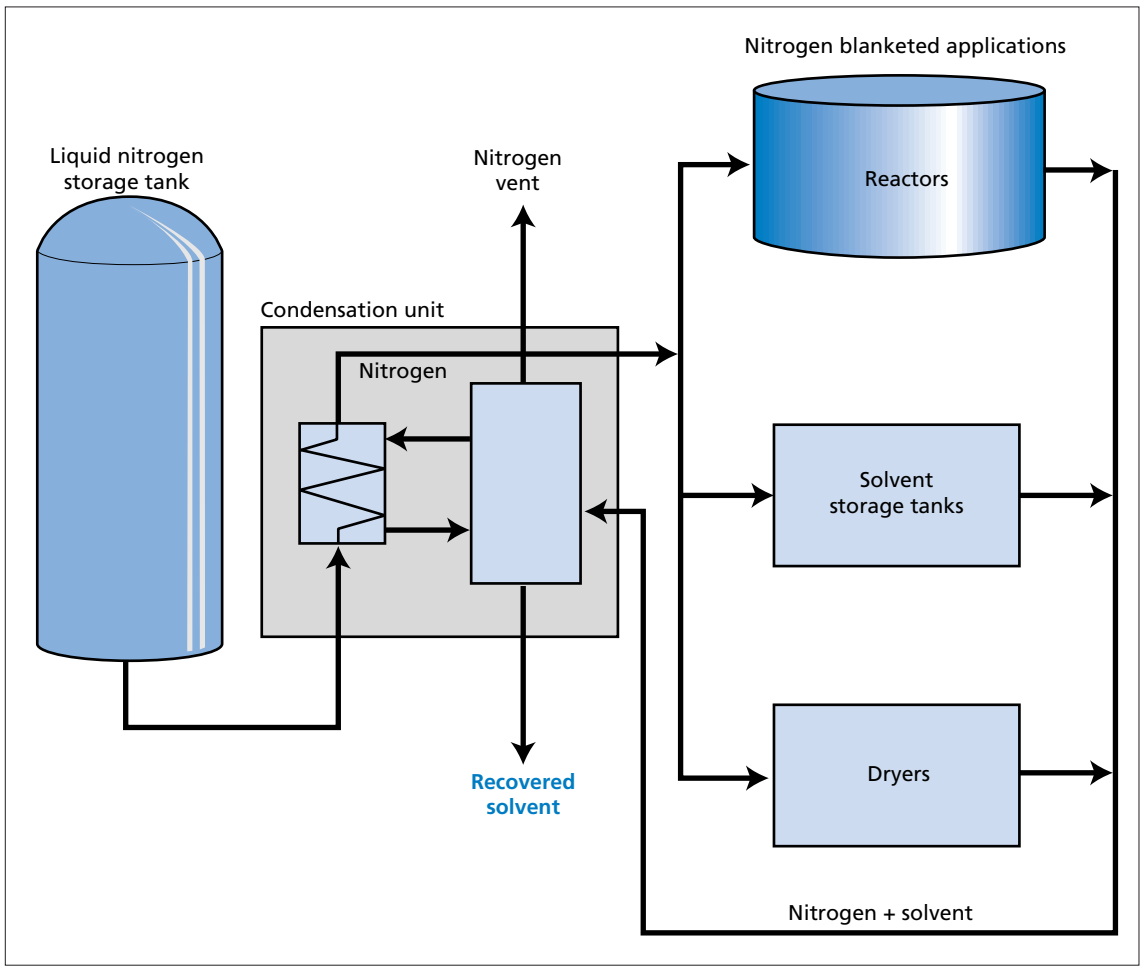


Fig 8 Solvent recovery system integral with a typical nitrogen blanketing operation

Various configurations of cryogenic recovery plant are available; an example is shown in Fig 9.

In Fig 9, the solvent-laden air passes first through a pre-condenser using chilled water or glycol. This stage maximises the efficiency of the main condensers by removing water and peaks of solvent vapour.

The main process condensers - two identical three-stage heat exchangers - gradually cool the process stream to the final condensation temperature. As the solvent vapours condense on the internal surfaces of these condensers, the liquid droplets drain to the bottom of the condenser. The cleaned nitrogen stream is then discharged to the atmosphere.

The alternate cool/thaw continuous cycle of the two-unit system is advantageous when high freezing point components result in frost accumulation on the heat transfer surfaces. All the water is thus removed from the recovered solvent. The switch-over occurs automatically when the differential pressure across the condenser reaches a pre-set point. The frozen condenser is thawed by a warm, sometimes steam-heated, fluid circulating around the inside of the heat exchanger. Any re-vaporised material is recycled to the working condenser inlet. The economisers reduce the system's refrigeration demand and maximise the refrigerant potential of the liquid nitrogen.

The recovered solvent should have a low water content and thus be potentially suitable for direct re-use. Mixtures may require further separation, either on-site or off-site by a specialised solvent

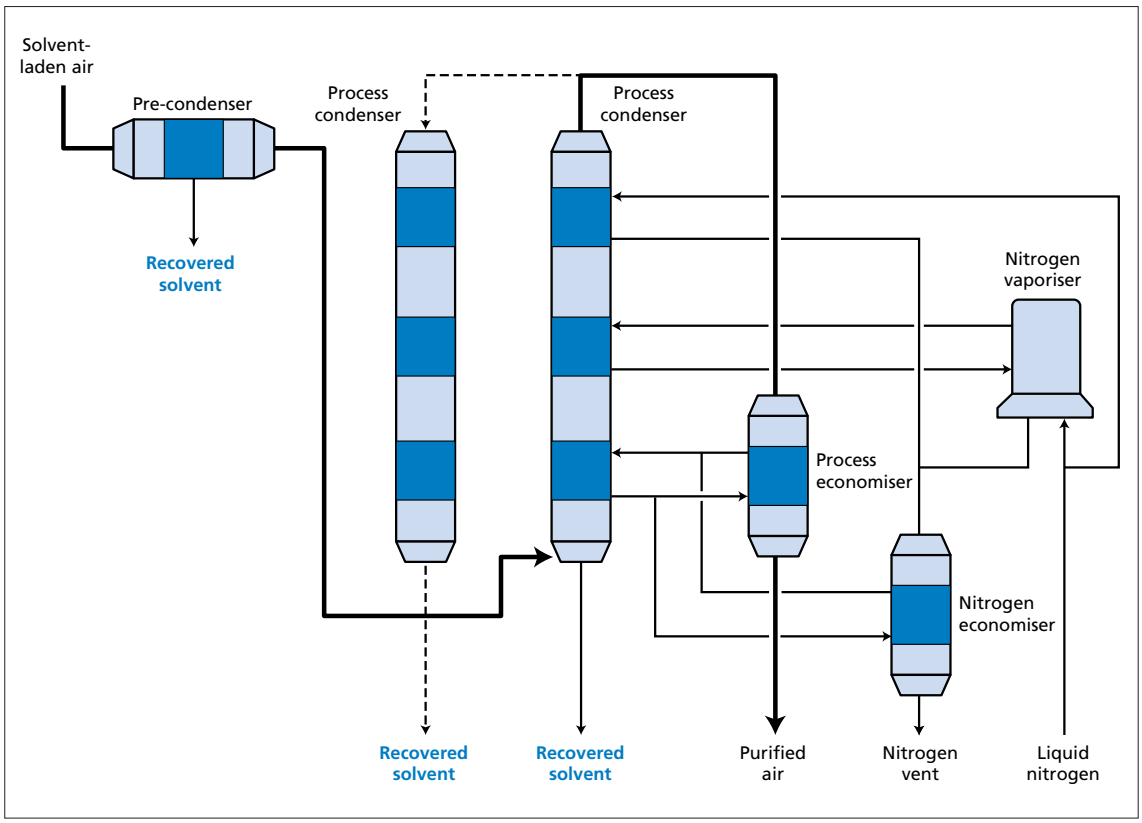


Fig 9 Example of a cryogenic recovery system

recovery company. Returning the recovered solvent back to process operating temperatures with a minimum of solvent vaporisation may require careful consideration.

The nitrogen supply should be located as close as possible to the solvent recovery unit to minimise cost.

4.5 CLOSED-CYCLE INERT GAS CONDENSATION

This type of equipment is designed for closed-cycle systems with high solvent vapour concentrations, eg solvent-laden gas streams from curing or drying ovens. A fixed volume of inert gas - generally nitrogen - is continuously recirculated around the oven and condensation unit. Solvent concentrations in the nitrogen stream of up to 40% volume for volume (v/v) are possible, thus reducing the relative size of the solvent recovery equipment. A proportion of the nitrogen/solvent vapour mixture is continuously drawn into the recovery module, where a series of heat exchangers cool and condense the solvent vapours.

A typical system (see Fig 10) for a drying oven - as used in the coating industries - has a main condenser which is mechanically refrigerated to as low as -40°C. Solvent from the exit stream from the main condenser is recovered in a solvent separator, while the nitrogen stream is subjected to a final non-cryogenic clean-up stage. Two streams exit from this stage: a cleaned nitrogen stream which is used to generate the nitrogen 'curtains' or oven seals; and a depleted mixed stream which is recycled.

Excess water present in the nitrogen/solvent stream can be removed prior to condensation by a molecular sieve unit. The feeders in the coating line are held at a slight positive pressure to avoid air infiltration and the escape of solvent-laden gas from the circuit. The whole system is in balance when the liquid nitrogen vaporised in the recovery module is equivalent to the nitrogen required at the 'curtains'.

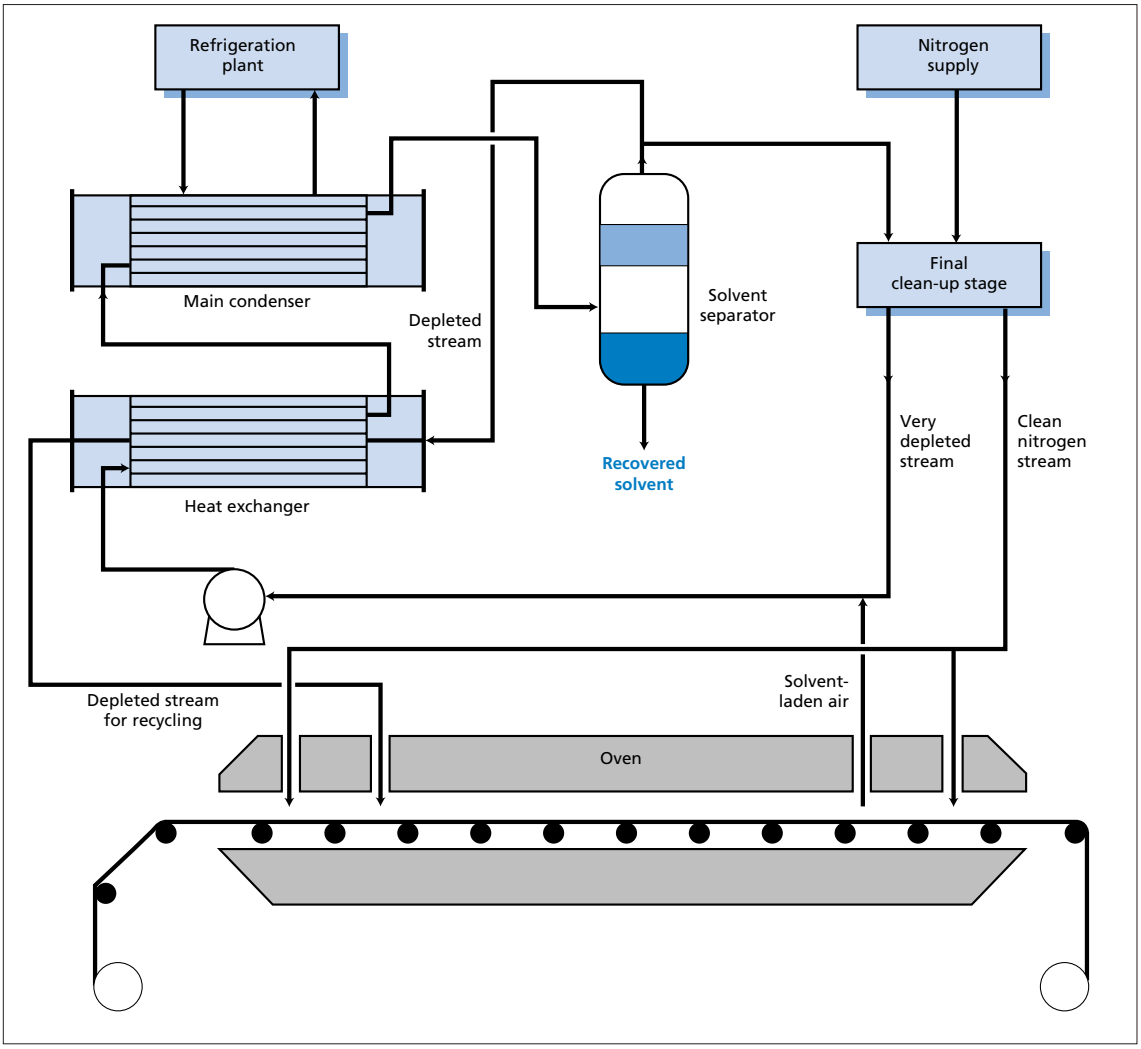


Fig 10 Typical closed-cycle inert gas condensation system

Use of an inert atmosphere can improve energy efficiency by up to 90% because heat losses are reduced by recirculating the oven atmosphere.

4.6 OPERATION AND INSTALLATION

4.6.1 Concentrations, flows and efficiencies

In general, condensation becomes more feasible with higher solvent concentrations and boiling points; concentrations of greater than 5 g/m^3 are preferable. At these concentrations, recovery efficiencies of 50 - 90% are achievable with refrigeration systems and 95 - 99.9% with cryogenic systems.

Condensers can handle a wide range of airflows, the main restriction being cost. For example, a cryogenic system with a flow of greater than 5 000 m^3/hr is unlikely to be cost-effective due to the expense of supplying the nitrogen required.

Basic heat exchange systems using refrigeration are designed for a maximum operating solvent concentration and airflow. For cryogenic systems, large fluctuations in solvent concentration and flow rate can be accommodated by controlling liquid nitrogen injection.

4.6.2 Utilities

The utilities required depend on the particular system.

Basic heat exchanger condensers require:

- a cooling liquid;
- electricity for fans, pumps and/or refrigeration plant.

Blanketing cryogenic systems require:

- liquid nitrogen;
- process coolant (if used);
- cooling water (if used);
- electricity for fans and refrigeration plant;
- steam to heat the fluid used to thaw frozen condensers (if required).

Inert gas cycle systems require:

- a nitrogen supply for
  - the inert atmosphere;
  - any emergency purges;
  - refrigeration (if used).
- electricity for fans and refrigerant plant.

An estimated 10 kg/hr of nitrogen cooling in the condensers is needed for each kilowatt of cooling required, but this depends on plant design, solvent type, etc. A typical inert gas cycle plant may use 1 - 2 tonnes/day.

4.6.3 Control and operation

Basic heat exchange condensers do not generally need dedicated control systems.

Cryogenic systems use standard programmable logic controllers to control nitrogen requirements for cooling. Additionally, on inert gas cycle systems, oxygen analysis is carried out to ensure that an inert atmosphere of less than 5% oxygen is maintained - for safety reasons - in the oven vent stream. If the oxygen content is too high, nitrogen is injected to restore the inert atmosphere. Automatic operation of the plant should be possible, provided alarms are set as required and routine checks are carried out by operators during each shift.

4.6.4 Maintenance

Maintenance requirements are generally limited to equipment with moving parts. If mechanical refrigeration is used, however, the high pressure compressors may demand significant maintenance. The maintenance of on-site liquid nitrogen tanks is usually the responsibility of the gas supplier.

4.6.5 Ease of retrofitting

Traditional condensation equipment can be readily retrofitted, with the heat exchangers being positioned nearby or on top of the relevant piece of equipment.

Cryogenic systems, which are usually only cost-effective on sites that already use nitrogen, may be skid-mounted. Such systems, which may replace any existing nitrogen vaporisers, should be installed near a liquid nitrogen source to minimise the length of cryogenic pipeline. Cryogenic condensation systems can either be retrofitted to existing plant or integrated into new plant.

In the case of drying ovens, inert gas cycle units are difficult to retrofit onto existing production plants; they are more suited to new plant.

4.7 COST FACTORS

The following factors influence the cost of a condensation recovery system.

4.7.1 Capital cost

Emission flow rate. This influences the overall size of the system.

Temperature reduction required. The greater the reduction in temperature to achieve the required level of solvent recovery, the more expensive the equipment. This is especially true for condensation temperatures well below 0°C because of the greater heat exchange surface required and the need for specialist equipment for low temperature operation.

Solvent mixtures. Multi-component solvent streams are likely to require more complex separation techniques.

Solvent solubility. The more soluble the solvent is in water, or other components present in the stream, the more complex the separation technique likely to be required.

4.7.2 Operating costs

Emission flow rate. This dictates utility requirements.

Cooling load. The greater the required cooling load, the more expensive the supply of cooling agent. This is especially true for condensation temperatures well below 0°C, as cryogenic systems can incur much higher costs unless nitrogen is already used on-site.

Solvent mixtures. More energy-intensive separation techniques are likely to be required.

Solvent solubility. The more soluble the solvent is in water, or other components present in the stream, the more energy-intensive the separation technique likely to be required.

4.8 SUMMARY

- Coolant/refrigerant condensation is a proven technology. Although widely used, it has traditionally been used for preliminary recovery prior to, for example, adsorption. Cryogenic systems are less widely used.
- Condensation techniques use various coolants and refrigerants, as well as cryogenic nitrogen.
- Concentrated emissions of high vapour pressure solvents are most suitable for recovery by conventional condensation, while all solvents - irrespective of their vapour pressure - can be recovered using cryogenic condensation.
- The economic feasibility of condensation depends on the temperature reduction required for effective recovery. Condensation temperatures well below 0°C can involve higher capital and operating costs unless nitrogen is already used on-site.
- Differential freezing points are likely to occur in the air stream due to the presence of water vapour and/or other components. Depending on the condensation temperature, this may lead to frozen material on the heat transfer surface and a subsequent reduction in condensation rate.
- Cryogenic systems are more likely to be economic where nitrogen is already used on-site.
- Changing from air to inert gas allows the airflow rate to be decreased and solvent concentrations increased without compromising safety. Changing the airflow in this way will reduce operating costs, thus making solvent capture and re-use more cost-effective.

5 ABSORPTION (SCRUBBING) AND ASSOCIATED TECHNIQUES

Absorption is a proven technology, with equipment suppliers having widespread experience of this type of equipment design and operation. Many such plants for solvent capture and re-use have been installed abroad. Most units in the UK have so far only been designed with disposal of the absorbed solvent in mind.

Absorption is a feasible recovery technique for solvents that are readily soluble in water or a high boiling point organic compound.

5.1 GENERAL PRINCIPLES

Absorption (scrubbing) involves mass transfer in a gas-liquid contacting device between a solvent vapour and a scrubbing liquid in which it is readily soluble.

Physical scrubbing, where the solvent does not react with the scrubbing liquid, is preferred for solvent recovery. Chemical scrubbing, where the solvent reacts with the scrubbing liquid, is used primarily for removal of the solvent without recovery. It is not considered here.

As a general rule:

- the height of the scrubber increases with required absorption efficiency;
- the cross-sectional area increases with increasing volumetric flow through the scrubber.

The driving force for absorption is the difference between the partial pressure of the solvent in the gas mixture and its vapour pressure in the liquid film in contact with the gas. Absorption does not occur unless this driving force is positive.

Close contact between the gas and the absorbing liquid is essential for effective absorption. This can be achieved by breaking up the liquid into small droplets or thin films, thus providing a greater liquid surface area for mass transfer.

For an individual solvent, the required scrubbing liquid circulation rate is determined largely by the airflow rate rather than its concentration in the air stream. The situation is similar in the desorption column, where the amount of steam required for stripping an individual solvent is controlled by the circulation rate rather than the solvent concentration. The scrubbing liquid circulation rate therefore tends to be governed by less easily absorbed components and the steam rate by those less easily desorbed.

As the solvent concentration in a gas stream increases, then so does its partial pressure. The scrubbing liquid is thus able to absorb more solvent while maintaining performance. At constant airflow, absorption systems therefore have a low sensitivity to variations in solvent concentration.

Higher gas solubilities also provide a greater driving force for more efficient absorption. Since lower temperatures correspond to lower vapour pressures and higher gas solubilities, absorption is enhanced at reduced temperatures.

Given a suitable choice of scrubbing liquid and plant design, absorption systems are capable of high solvent capture efficiencies over a wide range of solvents.

5.2 ABSORPTION (SCRUBBING)

A typical absorption/desorption system is shown in Fig 11.

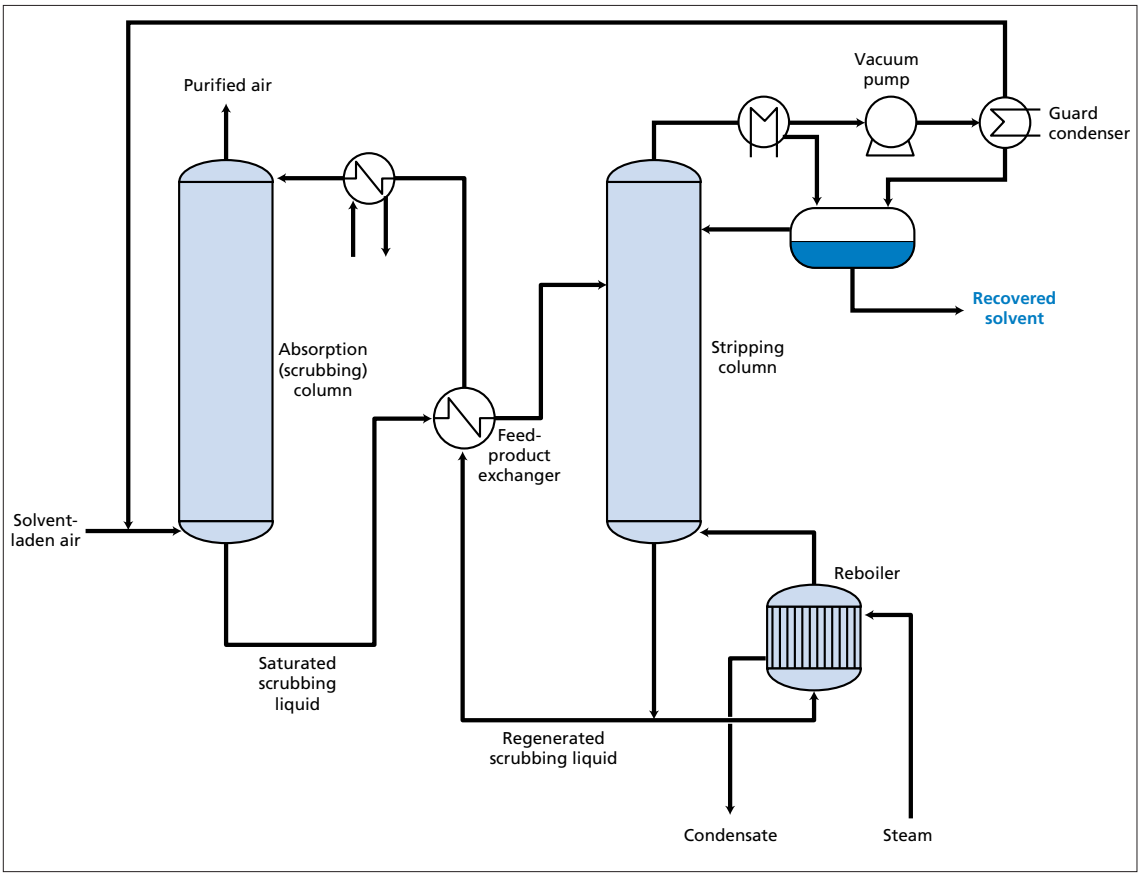


Fig 11 Typical absorption/desorption system

5.2.1 Scrubbing columns

Solvent-laden air usually flows upwards through packed, plate or spray columns against the flow of scrubbing liquid, ie in a countercurrent mode. The choice of column internals depends on several factors including:

- limitations on pressure drop;
- liquid and gas flow rates;
- cost.

The most common types of absorption equipment are packed or plate columns, or a combination of the two.

Packed column

A wide range of packing types is available, including rings, saddles and spheres. Recent developments include low pressure drop structured metal or plastic sheets, which offer greater contact surface areas than traditional packings. In addition, to improved absorption efficiencies, high gas and liquid throughputs are permitted.

Plate column

The plates can consist of either bubble caps, valved or perforated plates. The scrubbing liquid is introduced at the top and flows down from stage to stage, either through downcomer pipes or by dripping down through perforations in the plates.

A particular design of spray column uses an impingement baffle plate to achieve gas-liquid contact. The gas stream flows up through perforations in the trays, while the scrubbing liquid is sprayed at the underside of the trays (through the perforations) and allowed to flow back down the column. Gas-liquid contact and mass transfer are assisted by impingement plates located above each perforation. The gas flow impinges on these plates, creating additional turbulence and break-up of liquid films. As solvent vapours partition between the gas and liquid phases, the cooling effect enhances the transfer of vapour to the scrubbing liquid.

This column design results in a low pressure-drop, minimising power requirements for the fan.

5.2.2 Column fouling

Packed columns (unless fluidised) and, to a lesser extent, plate columns, are susceptible to fouling or plugging. This is due to:

- particulate matter in the inlet air stream;
- degradation or reaction products in the scrubber liquid stream.

These potential problems can be overcome by installing either a pre-filter to remove incoming particulate matter, or on-line filters to clean the recirculating liquid. A suitable stabilising agent is sometimes required for those organic liquids that have a tendency to degrade, eg ethers.

5.2.3 Scrubbing liquids

Water is the most commonly used scrubbing liquid. However, for water-based absorption, the solvent vapour must have adequate solubility in water at the operating temperature. For solvent vapours with limited water solubility, excessive quantities of water would be required and a low volatility organic liquid in which the solvent vapour has good solubility should be used instead.

Scrubbing liquids should satisfy the following criteria for economic, reliable and safe absorption.

Absorption/Desorption properties

Rapid absorption is preferred. Selective absorption may be required for some applications. Desorption temperatures and pressures depend on the type of solvent being scrubbed.

Vapour pressure

Vaporisation of an organic scrubbing liquid leads to additional VOC emissions, which may themselves require abatement. Liquids with low vapour pressures should therefore be selected. This criterion effectively rules out most potential scrubbing liquids with boiling points of less than 190°C for absorption processes operating at ambient temperatures.

Stability

Scrubbing liquids should be long-lasting in the recovery system. They need good thermal and chemical stability to avoid degradation during both absorption and desorption stages. Typical desorption temperatures range from 120 - 140°C.

Viscosity

The scrubbing column should be operated at as low a temperature as possible. However, many potential scrubbing liquids become viscous at low temperatures and do not spread well in packed columns. Ideally, the viscosity of the scrubbing liquid should not be significantly greater than that of water.

Safety/environmental data

Scrubbing liquids with high flash points and ignition temperatures are preferred. The liquid should also have a low toxicity and low odour.

Constant-boiling mixtures

The scrubbing liquid should ideally not form a constant boiling mixture with the solvent being absorbed as this creates problems during subsequent processing of the recovered solvent.

Suitable scrubbing liquids include:

- water;
- dimethylaniline;
- amines;
- higher glycol ethers such as polyethylene glycol and dibutyl ether.

5.3 DESORPTION

Desorption or regeneration of organic scrubbing liquids is usually carried out by steam stripping. This is carried out in a stripping column - usually a packed bed - designed to transfer the solvent from the liquid mixture back to the gaseous phase. The solvent is then condensed and collected. It can either be re-used directly in the process or separated into its component parts, eg by distillation. Steam stripping can be an effective technique for desorption, although water-soluble solvents may require complex separation techniques (eg distillation) for recovery.

In a typical absorption/desorption system (see Fig 11), the saturated scrubbing liquid from the bottom of the absorber is fed via a pre-heater into the desorption column. Here the solvent is stripped out under reduced pressure. The desorption column has steam injected directly into it from a re-boiler. The solvent is condensed in the condenser and collected. A guard condenser is fitted to maximise solvent recovery. Desorbers generally have large liquid flows with comparatively small vapour flows leading to tall columns with small diameters.

Inert gases, which pass through both condensers, are returned to the absorption column for venting to atmosphere. The regenerated scrubbing liquid leaves the bottom of the desorber and is recycled back to the absorber via the pre-heater and a final cooler (the absorber operates more efficiently at lower temperatures).

The stripping process is operated at reduced pressure to reduce the temperature required to strip the solvent from the liquor. This also minimises degradation of the organic scrubbing liquid, thus reducing the amount - and hence the cost - of liquid make-up required.

Degradation can cause particulate material to form within the scrubbing liquid. It may be necessary for on-line filters to be fitted to the absorber feed line, or for the liquid distributor to be carefully designed, so that any particles can be trapped. Fouling or blockage of the column internals are thus eliminated. The extent of degradation can be assessed by monitoring emissions from the absorber.

Water purged from scrubbing systems should be treated prior to discharge.

5.4 RE-USE OF SOLVENT

For an aqueous scrubbing unregenerated liquid, the most suitable solvent recovery method is likely to be distillation. However, the scrubbing liquid may be used directly in the manufacturing process, for example, in a polymer coagulation process where solutions of polyurethane in dimethylformamide (DMF) are extruded and coagulated in water. DMF vapour, which has a very high affinity for water, is scrubbed out of the extracted air using water in a two-stage (packed and sieve tray) column and the DMF-containing scrubbing liquid is returned directly to the process. The scrubbing efficiency is greater than 95%.

It may be possible to use a desorber to produce a solvent ready for direct re-use, particularly with single solvent streams. If two immiscible solvents are recovered, it may be possible to re-use them after separation in a phase separator.

If several solvents are captured by the scrubbing liquid or if the water content of the recovered solvent is too high, distillation may be required to obtain solvent in an acceptable form for re-use. Distillation can either be carried out on-site by the company concerned or off-site by a specialist solvent recovery company.

5.5 OPERATION AND INSTALLATION

5.5.1 Flows and efficiencies

The ability to vary three independent parameters to optimise plant performance gives absorption systems their versatility. Absorption efficiency can be improved by:

- designing the absorber for the maximum flow rate;
- increasing the scrubbing liquid circulation rate;
- reducing the gas stream temperature.

Desorption of high boiling solvents can be improved by adding more steam, although minimal quantities should be used to ensure that the recovered solvent contains the minimum amount of water.

5.5.2 Utilities

The utilities required include:

- power to drive the fans and pumps;
- cooling water or chilled water for the condensers;
- steam at about 120°C for the steam stripper;
- a vacuum system.

Given good heat exchange between the stripper input and output, the heat requirement for absorption is likely to be around 30 - 40 kg of steam per 1 000 m^3/hr of air. This requirement depends on:

- the type and latent heat of the solvent; compounds that are less easily absorbed and require more scrubbing liquid also require less steam per unit of scrubbing liquid for desorption;
- the solvent concentration in the air stream;
- the amount of reflux required.

In addition to utilities, make-up of the recirculating liquid is required. This depends on:

- the rate of degradation;
- any evaporative losses.

5.5.3 Control and operation

A programmable logic controller control system is typically used to control the operation of the plant automatically. A program is available which can predict the optimum operating parameters - ie circulation and steam requirement - for a given composition of the air stream. This is particularly useful where changes in the airflow and/or the solvent concentration are likely to be significant.

Action by plant operators is minimal - perhaps a brief check on the instruments during each shift - provided that automatic shut-down is fitted and alarms set up on the control system, eg for low liquid flow or loss of vacuum, etc.

5.5.4 Maintenance

Maintenance requirements are likely to be low. They are mainly limited to routine checks on the desorber vacuum system and equipment with moving parts.

5.5.5 Ease of retrofitting

Provided sufficient space is available, absorption systems can be relatively easily retrofitted to existing plant. Retrofits of existing absorption systems with improved structured packings and/or liquids can also be carried out to improve the operation of recovery equipment.

5.6 COST FACTORS

The following factors influence the cost of an absorption recovery system.

5.6.1 Capital cost

Emission flow rate. This influences the overall size of the system.

Recovery efficiency required. This influences the height of the scrubbing column.

Solvent type. This dictates the choice of scrubbing liquid.

Solvent mixtures. Multi-component solvent streams are likely to require more complex separation techniques.

Solvent solubility. The more soluble the solvent is in water, or other components present in the stream, the more complex the separation technique likely to be required.

5.6.2 Operating costs

Emission flow rate. This dictates utility requirements.

Solvent absorption efficiency. This influences the scrubbing liquid recirculation rate, and hence, pumping requirements.

Solvent loading. This dictates the rate of desorption and subsequent utility requirements, eg steam.

Scrubbing liquid degradation. Changes to the scrubbing liquid characteristics and solids build-up (due to high desorption temperature, chemical reactions, impurities in feed, etc) can lead to a high purge rate of spent liquid. This results in high treatment/disposal costs.

Ease of solvent desorption. High boiling point solvents require higher temperatures for desorption, but condense more readily.

Solvent mixtures. More energy-intensive separation techniques are likely to be required.

Solvent solubility. The more soluble the solvent is in water, or other components present in the stream, the more energy-intensive the separation technique likely to be required.

5.7 SUMMARY

- Although absorption (scrubbing) has been primarily used in the UK for emissions abatement, it is a feasible solvent recovery option.
- For scrubbing to be feasible, the solvent must be readily soluble in either water or a high boiling point organic compound.
- Absorption can take place in either packed, plate or spray columns.
- Organic scrubbing liquids are usually regenerated by steam stripping.
- Aqueous scrubbing liquids can be regenerated by distillation. But it may also be feasible to use them directly in the process.
- If the solvent is water-soluble, regeneration with steam may necessitate more complex separation techniques for solvent recovery.
- Degradation of the scrubbing liquid is a significant operational factor and may lead to high treatment/disposal costs for purged liquid. Degradation is caused by:
  - high desorption temperatures;
  - chemical reactions;
  - impurities in the feed.
- Provision must be made for the disposal of aqueous and other waste streams purged from the process.
- Absorption plants themselves require comparatively little floor space. Room is however required for any effluent treatment plant.
- Changing from air to inert gas allows the airflow rate to be decreased and solvent concentrations increased without compromising safety. Changing the airflow in this way will reduce operating costs, thus making solvent capture and re-use more cost-effective.

6 RECENT DEVELOPMENTS

Equipment suppliers are always looking for ways to improve the performance of their existing technology and to take account of recent developments. This Section outlines recent developments in solvent recovery techniques.

6.1 MEMBRANE PROCESSES

The use of very thin semi-permeable membranes has been developed in the USA over the last five to ten years. There are as yet no such installations for solvent capture in the UK. The solvent-laden air stream is passed over one side of the membrane and a vacuum applied on the other side. Driven by its partial pressure, the solvent diffuses through the membrane. This results in a more concentrated solvent stream on the vacuum side and condensation of most of the solvent-rich stream.

Efficient separation requires the membrane to be more permeable to the solvent vapour than to air and/or any other inert gas molecules in the air stream. Efficiency depends on the:

- selectivity of the membrane;
- pressure in the air space;
- pressure in the solvent-rich stream;
- extent to which solvent vapour has to be stripped from the air stream.

If the concentration of solvent vapour in the air stream is low, a high vacuum is required on the solvent-rich side of the membrane.

Few commercial applications have resulted from the extensive pilot plant studies carried out. This is mainly due to two significant technical problems. Firstly, the transmission fluxes attainable through most polymeric films of reasonable thickness are quite low. This means that extremely large membrane areas are needed to achieve a realistic capacity. Secondly, the selectivity ratios of most polymeric materials for solvents of commercial interest is modest. Many of the separations of interest are, therefore, not feasible with commercially available polymeric materials.

Due to the system's high cost, membrane processes are only applicable to low flow rates and solvent concentrations in the range of 1 - 10% v/v.

6.2 PLASTICISER/SOLVENT RECOVERY SYSTEM

Existing technology for the removal of plasticiser and solvent fumes from exhaust gases is being developed to enable recovery of both plasticiser and solvent.

In the initial stage, the exhaust gas is cooled to below 15°C to maximise the amount of solvent in the liquid phase. This is followed by an adsorptive recovery stage, which comprises specially designed levels of irrigation and contact medium. A final demisting stage completes the process. Additional recovery and/or treatment stages can be added to ensure that final emission levels are achieved.

Although the process can recover plasticisers efficiently, there are still problems in recovering the solvent. Some solvent is absorbed by the plasticiser, while plasticiser/solvent separation has proved difficult. However, the plasticiser/solvent mixture could be re-used, depending on the nature of the manufacturing process, eg in vinyl coating processes, such as wall or floor coverings.

6.3 ADSORBENT REGENERATION

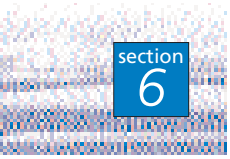
The use of a Brayton-cycle heat pump in an adsorption system reduces the energy cost for adsorbent regeneration by up to 25% compared with conventional systems. The integrated unit employs a vacuum pump turbo-compressor to regenerate a saturated adsorption bed. Hot inert gas from the compressor end of the unit desorbs solvent, which then passes into a chiller. The low temperature of the chiller is generated at the vacuum end of the unit. With cooling, the solvent condenses and can be recovered, while the inert carrier gas is ready to be compressed and heated again.

Another development designed to save energy costs during adsorbent regeneration involves the exchange of hot desorbate with water that is later flash-evaporated into low pressure steam. This steam passes through a thermo-compressor, raising both its pressure and temperature, and yielding new steam for adsorbent regeneration. The system should not only reduce energy costs by up to 50%, but should also conserve water for steam generation.

6.4 CONDENSATION SYSTEMS

Most developments in this area involve improvements to heat exchanger equipment. The aim is to maximise the use of the cooling load, and thus increase energy efficiency, by improving the equipment's heat exchange parameters. There are ongoing developments in compact heat exchanger technology which offer high performance and low cost heat transfer. See Energy Efficiency Best Practice programme Good Practice Guide 89, available from the Energy Efficiency Enquiries Bureau (01235 436747), for further information.

6.5 ABSORPTION SYSTEMS



The main development area is the improvement of column packings. Improved packings have three benefits:

- reduced pressure drop through the column (thus reducing fan power requirements);
- increased separation efficiency which increases the amount of solvent recovered from the air stream and/or reduces the reflux ratio required, minimising column operating costs;
- increased column capacity which either decreases the column diameter or increases the capacity of existing columns.

7 WHAT NEXT?

This Guide describes the capabilities of the three main technologies available for on-site solvent capture and re-use. It also indicates ways of improving the economics of each option by examining factors such as process airflows and solvent concentrations.

If you are thinking of cost-effectively reducing your company's VOC emissions you may benefit from answering the following questions.

- Have I investigated all possible ways of minimising solvent use?
- Could I re-use solvent directly in a process?
- Could I re-use solvent for some other operation, eg cleaning?
- Have I investigated waste exchange companies?
- Have I consulted solvent recovery companies?
- Can I make solvent recovery more economic by reducing airflows?
- Can I make solvent recovery more economic by increasing solvent concentrations subject to safety requirements?
- Should I change to single solvent operation to make recovery more attractive?
- Could newer solvent recovery techniques offer improved economics?

If, after reading this Good Practice Guide, you would like help with specific queries regarding solvent capture and re-use, or any environmental problem, please contact the Environmental Helpline on 0800 585794. The Helpline can provide you with:

- general information on solvent minimisation;
- Environmental Technology Best Practice Programme literature on solvent management, good housekeeping measures and, in the future, industrial case studies on solvent minimisation;
- details of waste exchange and solvent recovery companies.

Appendix 1

EQUIPMENT SUPPLIERS AND SOLVENT RECOVERY COMPANIES IN THE UK

A number of directories contain listings of solvent recovery equipment suppliers. These include:

- 'Process Engineering Index' published by Technical Indexes Ltd;
- 'Polmark' Directory (the European Pollution Control and Waste Management Directory) published by Ecotec Research & Consulting Ltd;
- Kompass published by the CBI;
- Environment Business Directory published by Information for Industry Ltd;
- Environment Industry Year book published by The Environment Press Ltd.

The Environmental Helpline (0800 585794) holds details on the above directories.

A list of UK equipment suppliers is given below. The list is not exhaustive and has been compiled from information currently available to the Environmental Technology Best Practice Programme. The listing of a supplier does not constitute an endorsement by the Programme of either its product or its competence, and neither does the omission of a supplier discriminate against its competence.

ADSORPTION/RECOVERY SUPPLIERS

AMEG (UK) Ltd
Tel: 01625 614675

Born Environmental Ltd
Tel: 01273 727374

Carbon Link Ltd
Tel: 01942 824240

Chematur Ltd
Tel: 01753 620056

Chemviron Carbon Ltd
Tel: 0161 6285000

CJB Developments Ltd
Tel: 01705 664911

Haden Drysys Env Ltd
Tel: 0121 7654040

Lurgi (UK) Ltd
Tel: 01483 730044

Neu Engineering Ltd
Tel: 0161 4564041

Norit UK Ltd
Tel: 0141 6418841

Rigidon (UK) Ltd
Tel: 01454 294006

Solviron Ltd
Tel: 01787 375004

Sutcliffe Croftshaw Ltd
Tel: 01942 275400

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CONDENSATION/RECOVERY SUPPLIERS

Air Products plc
Tel: 01270 506000

Alfa Laval Thermal Ltd
Tel: 0181 5680168

ETE Ltd
Tel: 01928 579283

BOC Gases
Tel: 01483 244315

ABSORPTION (SCRUBBING)/RECOVERY SUPPLIERS

Begg Cousland and Co Ltd
Tel: 0141 5565288

Born Environmental Ltd
Tel: 01273 727374

Eta Process Plant Ltd
Tel: 01889 576501

Lurgi (UK) Ltd
Tel: 01483 730044

Nairb Air Ltd
Tel: 0116 2608187

QVF Process Systems Ltd
Tel: 01785 817211

Spooner Industries Ltd
Tel: 01943 609505

Sulzer Chemtech Division
Tel: 01252 544311

The Environmental Helpline (0800 585794) will be able to provide you with information about specialist solvent recovery companies and waste exchange organisations.

Appendix 2
BIBLIOGRAPHY

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