



## **Chemical Solvent-Based Processes For Acid Gas Removal In Gasification Applications**



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## Introduction

Chemical solvent-based processes are well suited to the removal of acid gases from gasifier product streams. A combination of solvent choice and equipment design can be used to meet specific product and/or emission requirements. Examples will be reviewed of how specialty solvents can be used to meet different objectives, such as maximum selectivity for hydrogen sulfide (H<sub>2</sub>S) over carbon dioxide (CO<sub>2</sub>), enhanced carbonyl sulfide (COS) removal, or efficient total removal of CO<sub>2</sub>.

Gasifier streams also present unique challenges to the use of chemical solvent-based processes. Depending upon the gasifier feed stock, the solvent may become contaminated with a variety of species that impose an added burden on the operability of the treating process. Contaminants can be carried in with the gas and/or formed in-situ. A practical strategy for dealing with hydrogen cyanide, carboxylic acids, metal carbonyls and particulates will be discussed.

## Acid Gas Cleanup Technologies In Gasification Applications

All gasification processes include an acid gas cleanup step, regardless of the feed stock used or the ultimate use of the synthesis gas produced. Although several trials of hot (dry) gas cleanup have been conducted, all commercial acid gas cleanup today is carried out via cold (wet) systems. These fall into two broad classes: physical solvents and chemical solvents (and occasionally hybrids of the two). Both are proven technologies with many years of operating experience. Within these two broad categories there are many different products available [1,2].

Physical solvents, as the name implies, rely upon variations in the physical solubility of gases to effect separation. High solubilities of the contaminants are required for physical solvents to perform efficiently, and high partial pressures of those species provide the driving force for absorption. Union Carbide offers SELEXOL<sup>®</sup> Solvent, a proven physical solvent, via a licensed process with UOP. This was first used in gasification applications in the 1980's at Texaco/Coolwater and TVA/Muscle Shoals. More recently it has been selected for the Sarlux and api Energia projects. Its use in gasification applications has been described elsewhere [3] and will not be covered further here.

While hot potassium carbonate is used in CO<sub>2</sub> removal applications, the chemical solvents discussed here are amine-based and remove H<sub>2</sub>S and CO<sub>2</sub> via an acid-base reaction. Building on years of experience in natural gas, refinery, and synthesis gas plants, specially formulated chemical solvents have been developed to meet the various requirements of gasification plants. While experience in other applications has been invaluable in developing products for the gasification market, there have been several new challenges to overcome. This paper addresses the potential problems that can be encountered when using chemical solvents and offers practical solutions. The selection of an acid gas cleanup technology is influenced by many factors, including but not limited to:

- integration of synthesis gas cleanup with existing processes
- acid gas partial pressure
- selective versus total acid gas removal
- capital cost
- operating cost
- nature of feed gas contaminants

Each factor will be described briefly. In-depth discussion is presented concerning the nature of contaminants and their impact on process selection and unit operation.

## **Process Integration**

The popularity of gasification as an economical and efficient disposal method for refinery bottoms presents interesting possibilities for the integration of acid gas cleanup systems with existing sulfur removal and recovery equipment. All refineries have amine-based systems for handling H<sub>2</sub>S. Utilizing any excess capacity in existing units offers the ability to lower capital costs for a new gasifier. As will be discussed, cross contamination of amine solutions is a real concern for such a scenario. However, with proper anticipation of potential contaminants, both from the refinery and from the gasifier, plans can be made for the pre-treatment of gas streams and/or the reclamation of contaminated solution.

There are also ways to create additional capacity in existing desulfurization equipment without capital expenditure. Specially formulated treating solvents are available that operate at higher concentrations than generic monoethanolamine (MEA) or diethanolamine (DEA). These same solutions may be appropriate for use in an integrated gasifier complex.

## **Acid Gas Partial Pressure**

With physical solvents, acid gas partial pressure provides the driving force for absorption. The higher the pressure, the lower the required solvent circulation rate to effect separation. This improves operating economics for physical solvents.

With chemical solvents, partial pressure is the driving force for mass transfer. Fewer absorber stages are required to effect separation at higher pressure. This reduces the capital requirement for chemical solvents.

The partial pressure of CO<sub>2</sub> and H<sub>2</sub>S also affects a solvent's ability to selectively remove H<sub>2</sub>S while slipping CO<sub>2</sub> into the treated gas stream. IGCC applications require selective removal of H<sub>2</sub>S and maximum CO<sub>2</sub> slip. A solvent with superior slip characteristics offers considerable advantage because more gas will be available to produce power in the turbine.

## **Selective Acid Gas Removal: IGCC**

When the raw material fed to the gasifier contains sulfur, the principle sulfur species in the raw synthesis gas are H<sub>2</sub>S and COS. For subsequent combustion in a gas turbine, the level of sulfur species must typically be less than 50 ppm. From the perspective of overall energy efficiency, the slip of CO<sub>2</sub> through the acid gas removal unit should be as high as possible. Methyldiethanolamine (MDEA) is often cited as the solvent of choice in this application, giving good sulfur removal and reasonable CO<sub>2</sub> slip. Several authors have addressed the mechanism by which MDEA selectively absorbs H<sub>2</sub>S [4,5].

Figure 1 shows a simplified process flow diagram for chemical solvent-based acid gas treating. Cooled synthesis gas enters the bottom of an absorber where it contacts an aqueous chemical solvent solution. The treated gas exits the absorber and continues to the next processing step, which is the gas turbine in IGCC applications. Cool lean solution enters the top of the absorber and counter-currently contacts the synthesis gas using trays or packing, absorbing acid gas contaminants as it passes down the column. Warm rich solution leaves the bottom of the absorber and is routed to a regenerator. Steam stripping is used to remove acid gas from the solution. This results in a concentrated acid gas stream which can be fed to a Claus sulfur recovery unit. The hot lean solution is then cooled prior to returning to the absorber. A lean/rich cross exchanger is used to reduce the sensible heat load on the regenerator reboiler.



## Total Acid Gas Removal: Chemical Production

In applications where the gasifier product is destined to be used as a chemical feed stock, complete removal of carbon dioxide and sulfur species is required. CO<sub>2</sub> specifications of less than 100 ppm are typical. CO<sub>2</sub> removal can be accomplished at the same time the sulfur is removed (total acid gas removal) or a portion of the synthesis gas can be treated for further CO<sub>2</sub> removal after the H<sub>2</sub>S has been selectively removed. This scheme has the advantage of concentrating the H<sub>2</sub>S from the first treater for feed to a sulfur recovery unit. In some chemical feed stock applications it proves economical to generate the synthesis gas from sulfur-free raw materials, such as natural gas, so that only CO<sub>2</sub> removal is required.

In the past, MEA and inhibited MEA have been used for this application. Specialty solvents are now available which offer significantly better operating efficiency versus MEA. An example using a synthesis gas stream with moderate CO<sub>2</sub> content is shown in Example 2.

Significant efficiency is gained from the use of formulated MDEA-based solvents by operating at 50 wt% in lieu of lower concentrations for generic amines. Industry experience indicates that primary amines cannot be operated at higher concentrations without increasing the potential for corrosion. Operating at higher strength reduces the solvent circulation which reduces the sensible heat load on the regenerator. Further energy efficiency is gained because the heat of reaction between CO<sub>2</sub> and tertiary amines is lower than the heat of reaction with primary amines.

### Example 2: Efficient CO<sub>2</sub> Removal\*

Feed Gas: 153,000 Nm<sup>3</sup>/hr (137 MMscfd)  
2620 kPa (380 psia); 40°C (104°F)  
CO<sub>2</sub>: 9.3 mole%

Solvent	18 wt% MEA	50 wt% UCARSOL <sup>®</sup> Solvent CR
Circ. Rate, m <sup>3</sup> /hr (gpm)	667 (2940)	381 (1680)
CO <sub>2</sub> Specification, ppmv	100	100
Reflux Ratio, mole/mole	2.5	1.25
Reboiler Duty, Gcal/hr (MMBtu/hr)	42.1 (163.3)	23.8 (94.6)

\*Based on 30-tray absorber, 17°C (30°F) L/R exchanger approach.

## Capital and Operating Costs

In a typical gasification project, the cost of the acid gas removal system represents only a small proportion of the overall project cost. However, the choice of cleanup technology and design of the acid gas removal unit has long term consequences for plant reliability and cost of operation. These can have a significant impact on the ultimate viability of the project. This paper will not address capital and operating costs directly but, instead, will focus on operational difficulties that can occur when chemical solvents are chosen for the acid gas cleanup. Practical solutions for these potential problems are presented.

## Feed Contaminants

Gasifier synthesis gas contaminants, other than H<sub>2</sub>S and CO<sub>2</sub>, fall into four main categories: metal carbonyls, COS, foam promoters and foulants, and carboxylic acids and their precursors that form heat-stable amine salts.

## Metal Carbonyls

Iron and nickel carbonyls present an interesting problem. They are only partially soluble in aqueous solutions, so consideration has to be given to the potential impact on downstream turbine blades. If the level of carbonyls removal by chemical solvents is adequate, they can then be removed from the working solution via particulate filters. In this event, provision for handling of potentially hazardous filter cake has to be made. Experience indicates that moist filter cake presents no airborne hazard, and protective clothing is adequate to protect workers from dermal contact.

If the anticipated level of metal carbonyl contamination in the treated synthesis gas is not acceptable using chemical solvents, it may be advisable to use a physical solvent to achieve total removal of the metal carbonyls.

## COS Removal

Local environmental regulations typically control the level to which sulfur must be removed. In cases where very strict effluent levels are required, COS hydrolysis may be recommended upstream of the acid gas removal unit. This step converts all but a few ppm of the COS to hydrogen sulfide. Solvent choice also plays a part in the decision for/against COS hydrolysis as different solvents are able to remove COS to different levels under given conditions. Example 3 demonstrates how one specially formulated MDEA-based solvent is able to enhance COS removal while maintaining most of its CO<sub>2</sub> slip.

### Example 3: Enhanced COS Removal\*

Feed Gas: 175,000 Nm<sup>3</sup>/hr (157 MMscfd)  
2690 kPa (390 psia); 40°C (104°F)  
CO<sub>2</sub>: 10.6 mole%  
H<sub>2</sub>S: 0.6 mole%  
COS: 30 ppmv

Solvent	MDEA	UCARSOL <sup>®</sup> Solvent HS-101	UCARSOL <sup>®</sup> Solvent HS-104
Circ. Rate, m <sup>3</sup> /hr (gpm)	192 (845)	184 (810)	200 (879)
CO <sub>2</sub> Slip, % of inlet	78	80	76
COS Removal, % of inlet	10-20	10-20	40-50

\* Based on 50 wt% solutions, 10-tray absorber.

The COS removal performance of specially formulated solvents may be sufficient to avoid the installation of a COS hydrolysis reactor. A penalty is paid, however, in reduced CO<sub>2</sub> slip. Upstream COS hydrolysis is probably preferred in facilities which must meet stringent total sulfur emission levels.

## Foam Promoters

A significant number of operational problems associated with wet solvent systems can be traced to solvent contamination by soot/particulates, iron sulfide, tars, or surface active species such as hydrocarbons. For the most part these are introduced unintentionally with the synthesis gas. Adequate and reliable pre-treatment of the synthesis gas is the best way of minimizing contamination of the acid gas cleanup solvent. In a typical gasification process the hot synthesis gas exits the gasifier and is passed through a series of waste heat boilers, quenches, and water washes, to recover sensible heat as steam, and remove the soot, tars, and higher boiling hydrocarbons that are unavoidably formed during the gasification process. A wide variety of water washes are employed [6]. These washes are not always as efficient as expected, particularly with very fine aerosols or particulates. Oftentimes they simply malfunction or are under-designed for startup or upset conditions. Union Carbide has developed recommendations for dealing with each contaminant, based on experience in hundreds of chemical solvent-based treating units.

Clean, uncontaminated treating solutions have a very low tendency to foam. This is confirmed by reports in the literature [7] as well as by Union Carbide's field experience. It has also been confirmed that the addition of sparingly soluble contaminants, such as high molecular weight hydrocarbons, tars, or lubrication oils, increases the foaming tendency. Operationally, foaming can lead to increased solvent losses and off-specification treating.

An activated carbon filter is recommended in the chemical treating system to purify a 10-20% slip-stream of cool lean solution. This is usually adequate to take care of chronic contamination problems. Anti-foam agents are administered as needed to suppress foaming during acute contamination episodes. Administering anti-foam to a system on a routine basis should not be required and will shorten the life of the activated carbon bed, but injection systems should be set up so that it can be added quickly when necessary.

The level of metal carbonyls and particulates in the treating solution can be reduced by filtration, and filter suppliers recommend a variety of operating schemes and filter types. To avoid contamination of the regenerator, rich-side filtration is recommended, though worker safety must be addressed when H<sub>2</sub>S is present in the rich solution. At a minimum, 10-20% slipstream filtration should be coupled with carbon filtration of the cool lean solution. The more filtration of the working solution provided, the better the solvent will perform and the more trouble-free the unit operation.

## Heat Stable Amine Salts

Perhaps the most significant contaminants are Heat-Stable Amine Salts (HSAS). These are formed when the basic solvent reacts with a relatively strong acid. HSAS are one of the more intractable results of contamination. Although degradation of the amine can also lead to their formation, HSAS precursors are usually introduced with the synthesis gas.

The introduction of any relatively strong acid into the amine system will result in the formation of HSAS, a reduction in pH, and deactivation of the solvent from an acid gas removal perspective. If instead of reacting with a weak acid in the synthesis gas, such as H<sub>2</sub>S (which has a pK<sub>a</sub> of 7.05 at 20°C), the solvent reacts with a stronger acid (pK<sub>a</sub> <6), it proves impossible to reverse this to any great extent at normal stripper conditions. The resulting salt is said to be heat-stable because of this inability to reverse the reaction. For example, with formic acid (pK<sub>a</sub> 3.76), the amine formate salt is readily formed but cannot be reversed:





Low levels of acids or acid precursors are absorbed into the solvent from the synthesis gas being treated. Since they can be lost from the system only via mechanical losses, not by vaporization, they tend to steadily accumulate. Impurities in the gasifier feed stock can lead to the introduction of HSAS in the solvent, but one of the major sources of HSAS may be carbon monoxide (CO), which can lead to the creation of formate anions.

While the partial pressure of CO in synthesis gas can vary widely, it is true to say that it is always significantly higher than that encountered in other gas treating applications. One unfortunate result of this is the generation of formates, most likely as a consequence of the following simple reaction [8]:



Although the rate of reaction (2) is very slow, it is irreversible and formate HSAS will steadily accumulate in solution over time. For example, in one system treating synthesis gas with a CO partial pressure of 10 bar, formate anions build at a rate of 150 ppmw/day. In addition to the CO partial pressure, the rate of formate formation via this route increases as a function of increasing pH and temperature. Unfortunately, reducing any of these three parameters is either impractical or counter-productive to the main purpose of the cleanup unit, which is removal of H<sub>2</sub>S and/or CO<sub>2</sub> to very low levels.

Other potential routes to formate from CO are via amide or formate ester intermediates, particularly in total CO<sub>2</sub> removal systems, but these routes need further verification before they are proven. Fortunately amides themselves do not pose any significant corrosion problems compared to formate anions. Suffice it to say that whatever the mechanism, formate accumulation is an unavoidable consequence of treating synthesis gas and provision has to be made ahead of time to control, mitigate, and ultimately remove formates from the system.

Two nitrogen based contaminants, ammonia and hydrogen cyanide (HCN), are often encountered and are absorbed from the synthesis gas by chemical-based treating solutions. The various water-wash and quench systems upstream of the acid gas cleanup unit should remove the majority of these species but a small amount will still get through to the treating system. Ammonia does not lead to HSAS formation and can be easily removed by purging regenerator reflux water. However, cyanide incursion is a more serious problem since it can be quite corrosive, forming soluble ferrocyanide complexes, as well as acidic anions which form HSAS. HCN itself is a weak acid, but it reacts in a basic solution and converts to stronger acids that do form corrosive heat-stable amine salts.

### **Problems Associated With Heat Stable Amine Salts**

The greatest problem posed by HSAS is the increased potential for corrosion [9, 10]. Although there is no definitive explanation for HSAS corrosion, one promising hypothesis is that as the anion level increases so does the level of undissociated acid in equilibrium with the anion [11]. The undissociated acid is the active species promoting corrosion by catalyzing the cathodic reaction. If the pH and acid loadings (HSAS as well as acid gas) are known, the level of undissociated acid can be calculated, taking into account the amine and acid pK<sub>a</sub> values. This exercise reveals that the most corrosive HSAS are those associated with the medium strength acids (e.g. formic, acetic, and glycolic) rather than the stronger acids, since the former lead to the greatest concentration of undissociated acid in solution. Higher temperatures increase the concentration of undissociated acids, making hot lean areas of the treating unit particularly susceptible to corrosion.

By themselves, the typical HSAS encountered in gasification applications (formates and thiocyanates), being soluble and ionic in nature, do not promote foaming. However, by increasing corrosion rates, they can increase the particulate load and thus indirectly cause foaming.

## HSAS Control and Removal Strategies

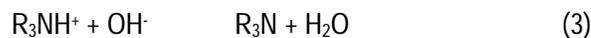
The best solution to HSAS problems is to prevent the precursors from entering the amine system in the first place. The pre-wash systems discussed earlier should achieve a good degree of reduction. However, with unavoidably high CO partial pressures, pre-washing will not eliminate all HSAS problems. Options have to be available to control and treat HSAS problems when they do occur.

One apparently simple solution to increased HSAS levels is to purge contaminated solvent and makeup with fresh material. Unfortunately this significantly increases the operating costs of the cleanup unit. The biological oxidation demand on the waste treatment system is also increased when any contaminated solvent solutions are sent to the sewer. With new discharge limits imposed on waste treatment systems, this is not always a feasible proposition. Deliberate purge-and-makeup is thus neither an economically nor environmentally attractive option.

In MEA systems HSAS problems can be taken care of by the use of a reclaimer: a semi-batch distillation process operated at atmospheric pressure. A slip stream of MEA solution is fed to the reclaimer, and water and MEA are stripped overhead, leaving behind MEA degradation products, HSAS and, if used, corrosion inhibitors. This approach cannot be applied to systems running on MDEA or MDEA-based formulated products since the atmospheric boiling points of MEA and MDEA are 171°C (340°F) and 247°C (477°F) respectively. Significant thermal degradation would result if MDEA was reclaimed at atmospheric pressure. The costs and operating complexity associated with setting up an on-line vacuum reclaimer are considered prohibitive. Therefore, to fill the need for on-line reclaiming of MDEA-based specialty products, Union Carbide developed an electro dialysis process. This technology, commercially known as the UCARSEP® Process, has been successfully used in the field [12]. By coupling this with a strategy of HSAS control via neutralization, the advantages of this technology are further strengthened.

## HSAS Control Via Neutralization

One proven means of mitigating the effects of HSAS is to neutralize using a stronger base than the amine in question. This will raise the system pH, deprotonate the amine, and render it available again for acid gas removal purposes. The overall effect is shown below:



There is a lot of evidence in the literature for the benefits of neutralization as a means of controlling HSAS problems [9, 10]. More importantly, this is also supported by industry experience [13]. Caustic has been used as the strong base, but this is not the most suitable choice since sodium salts are not always soluble in chemical solvent systems. Caustic can also be an unintentional source of chlorides and, if not administered carefully, can lead to concerns with over-neutralization, precipitation/fouling, and stress corrosion cracking. A proprietary neutralizing agent has been developed by Union Carbide for use in the many situations where caustic proves unsuitable.

Union Carbide's experience has been that without neutralization, HSAS anion levels of up to 10,000 ppmw can be tolerated without significant corrosion. Since neutralized salts are less corrosive than the corresponding amine salts [9, 10], a higher level of anions is permissible if neutralization is practiced. Trouble-free operation with anion levels as high as 50,000 ppmw

is possible with judicious and regular neutralization to maintain the HSAS levels at ~1 wt%. Any system's corrosion-free contaminant level is influenced by the type of anion as well as its concentration. Although it can be misleading to generalize, it has been found that a contamination level of <30-40,000 ppmw anions (coupled with <1-2 wt% HSAS) is a practical upper limit. Neutralization is thus a very pragmatic and effective solution to the HSAS problem. For example, after implementing a program of neutralization, one user reported greatly improved operation: comparing the six month period before and after treatment, the number of heat exchanger washes was reduced from four to none, the number of absorber washes went from ten to none, and the number of filter changes was reduced from sixteen to four [10].

Being able to operate safely at higher anion levels has the added benefit of extending the time before solvent reclamation is required. Depending upon the relative rate of incursion and loss, the need for reclamation may be averted completely. As the level of contamination increases, mechanical solution losses, which are fairly constant if viewed over a long enough time frame, account for larger and larger contaminant losses. This increases the time between reclamation and decreases the amount of salt that has to be removed when reclamation is required. Taking advantage of unavoidable system losses in this way is far removed from setting up a deliberate purge-and-make-up procedure to control HSAS.

## **Electrodialysis Technology for HSAS Removal**

Electrodialysis (ED) has been widely used in the water treating industry for many years. Recognizing that it has beneficial characteristics for salt removal and fits very well with neutralization, Union Carbide adapted it to the unique conditions encountered in acid gas cleanup [14].

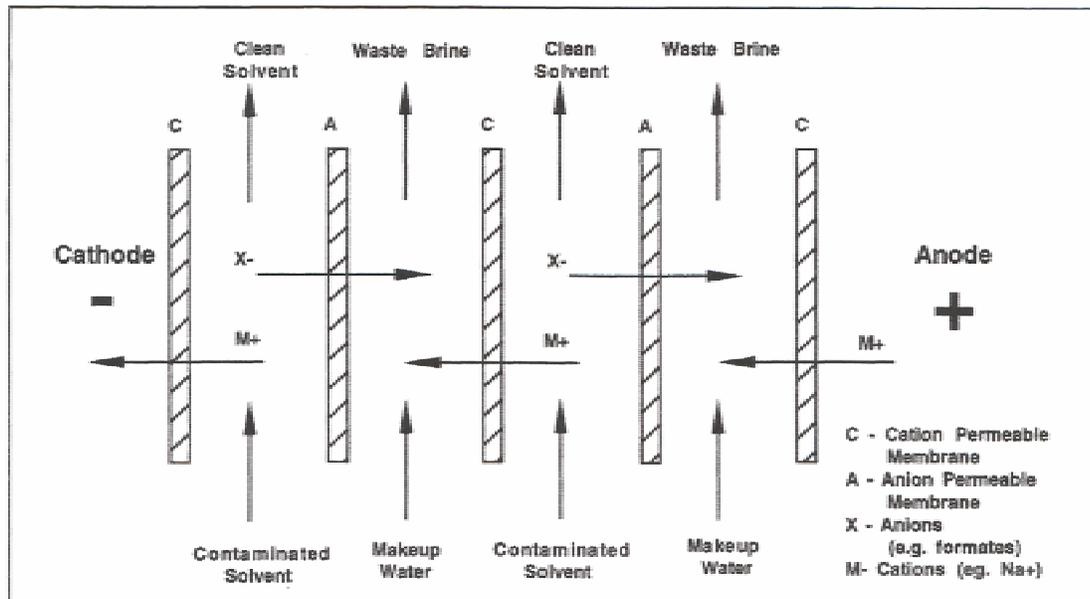
ED is a separation process in which ion-permeable membranes are placed in an electric field to facilitate the removal of substances that ionize in solution. These semi-permeable membranes contain electrically charged functional sites chosen so that they are selective and allow the passage of either anions or cations, but not both. By correct sequencing, anions and cations can be extracted from one solution into another as shown in Figure 2. The membranes are sequenced such that when the solvent enters the channel between an anion- and cation-permeable membrane, the anions move towards the anode through the anion-permeable membrane, and the cations move towards the cathode through the cation-permeable membrane. On the other side of both membranes an aqueous brine solution flows and the transported ions are collected and swept out of the system for disposal.

The technology can be tailored to the specific requirements of any treating unit to provide a dedicated on-site HSAS removal capability. Typically several hundred cell pairs are required, but the exact number and membrane area needed are governed by the required salt removal duty. However, the overall process is very compact and the space requirement is small.

For systems where a permanent unit cannot be justified because the contamination problem is periodic or controllable through judicious neutralization, a mobile ED unit capable of removing up to ~0.2 mole/sec of salts has been built. The unit can be brought on-site and cleanup accomplished on-line in a minimal amount of time. Only a small slip-stream of contaminated lean solution is required (typically <1 % of circulation) and experience has shown that the operation of the treating unit is unaffected by the reclamation. The process is fully automated and operates 24 hours a day. Process and utility hookups are simple, and power consumption costs are minimal. A source of good quality water for brine make-up is required. Water has to be added to the brine loop to maintain a constant salt concentration in the brine, but water is neither added nor taken out of the solvent itself.

One of the benefits of the ED process is that the aqueous brine stream is considered to be biodegradable and non-hazardous. The brine is homogeneous, has a pH typically in the range of 9-10, and does not require any post-treatment before discharge to a conventional waste water treatment system (or routing through the gasifier itself). Unlike conventional ion exchange absorption processes, the volume of brine is simply proportional to the amount of salt removed since flushing or back-washing with rinse water and regeneration chemicals are not required [15]. In this way the hydraulic load and biological oxygen demand on the wastewater treatment system are minimized.

**Figure 2 • Principle of Solvent Reclamation Via Electrodialysis**



**Summary**

Chemical solvent-based processes are well suited for acid gas cleanup of gasifier product streams. The combination of a specially formulated solvent and well designed equipment can be used to meet a variety of product gas and/or emission requirements. Superior selectivity for H<sub>2</sub>S over CO<sub>2</sub>, enhanced COS removal, and efficient total CO<sub>2</sub> removal can be achieved more economically with specialty solvents than with corresponding generic amine solutions.

In providing the intimate contact necessary to meet H<sub>2</sub>S and CO<sub>2</sub> specifications, there is more than adequate contact for a chemical solvent to absorb other contaminants from the gas. Solvent contamination can be directly linked with increased levels of foaming, fouling, and corrosion. These symptoms in turn result in increased solvent losses, off-specification operation, and possible equipment failure/replacement. The extent to which the acid gas cleanup systems can handle these diverse contaminants, or to which provision is made to remove them upstream, will have a great impact upon the operability of the unit and overall plant reliability. Since reliability is a key concern in gasification applications, it is imperative that these issues are taken into account at the design stage.

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