thiocyanate reagent and a ferric alum indicator. Unfortunately, the method is subject to many interferences, including aluminum, beryllium, iron, potassium, sodium, ammonia, phosphate, and sulfate.

Two other titrimetric methods operate on the basis of adding titrant and detecting the first excess of titrant, thus indicating that all fluoride has reacted. One of these titrations uses calcium nitrate titrant; the other is the lead nitrate–hydrochloric acid reaction. The calcium method would not work with developers that contain large amounts of calcium, and the lead nitrate method is subject to the same list of interferences as ferric alum, noted previously.

Probably the most promising of the titrimetric procedures is the thorium nitrate titration with sodium alizarin sulfonate indicator. This method is only partially applicable to penetrant testing materials because of certain interferences. Even in small quantities, phosphate is a serious interference, causing a red color, which hides the end point completely. Phosphorous is often a constituent of test materials. Another interference is any acid insoluble solid, for example, many developer ingredients; these completely mask the end point.

## Photometry for Fluoride Analysis

A third general procedure that has been used is the reaction of the dissolved fluorides with some color forming reagent to produce a solution capable of being measured photometrically. In these tests, the depth of color is proportional to the fluoride concentration. Probably the best known of these analyses is the method of ASTM D 1179, which uses a compound of sodium, 2-(parasulfophenylazo), 1,8-dihydroxy, and 3,6-naphthalene disulfonate (SPADNS). This material, dissolved in water and mixed with a zirconyl chloride hydrochloric acid mixture, is bleached by fluoride. The amount of bleaching is then measured photometrically.

The ASTM D 1179 method is subject to interference from aluminum, iron, phosphate, and sulfate, all of which may be present in penetrant test materials.

## Fluoride Analysis with Fluoride Electrode

A fluoride specific ion electrode can eliminate most of the problems of the other techniques and has been used for some fluoride measurements. When the electrode is immersed in a solution containing fluoride ions, an electrical potential develops between the fluoride electrode and a reference electrode also in the solution. The potential decreases with increasing fluoride content. Further, the specific ion electrode responds only to the fluoride ion and the hydroxide ion if hydroxide is in greater concentration than fluoride. The proportion can be controlled easily by adjusting to a lower pH.

Fluoride ions can be lost through its complexing with cations such as aluminum (III) ($Al^{3+}$), ferric ion or iron (III) ($Fe^{3+}$), or silicon (IV) ($Si^{4+}$). This interference can be eliminated by the addition of an agent such as citrate, (cyclohexylenedinitrilo) acetic acid (CDTA), or ethylenediaminetetraacetate (EDTA). Another limitation to fluoride measurements with the specific ion electrode is that the response is not directly to the concentration but to the ion activity. The activity of an ion in solution is modified by other ions in solution. If the fluoride present is ionized and the pH is properly controlled, there are essentially no interferences with this method.

## Ion Chromatography for Fluoride Analysis

Another method for fluoride testing is ion chromatography. After the sample is prepared by calorimetric bomb combustion, a filtered aliquot is injected into a stream of carbonate/bicarbonate eluent and passes through a series of ion exchangers. The anions of interest are separated on the basis of their affinities for a strongly basic anion exchanger. The separated anions are measured by conductivity. They are identified on the basis of retention time as compared to standards. Quantitation is a measurement of peak area or peak height.

# Part 3

# Control of Pollution from Liquid Penetrant Waste

## LIQUID PENETRANT CONSTITUENTS CONTRIBUTING TO WASTE POLLUTION

Penetrants may contain a considerable variety of chemical compounds. Among those fairly widely used are petroleum products ranging from relatively low boiling range solvents to high boiling oils. Other generic agents are nonionic and surface active. Anionic surface active agents are less commonly used because their presence in penetrants has disadvantages that outweigh their excellent surface active properties and, perhaps more importantly, biologically degrade more easily. Other constituents present in significant proportions may be alcohols, glycol ethers, and esters, to name but a few. Finally, penetrants contain red and fluorescent dyes. These dyes are the most troublesome contaminants because they are visibly detectable at low concentration and can be traced to their source (Figure 8).

## REDUCING LIQUID PENETRANT PROCESS WASTES

The following chemical engineering or penetrant processing steps are suggested to reduce the amount of penetrant product residues in the waste effluent from penetrant testing processing systems.

1. Minimize the amount of penetrant applied. Electrostatic and airless spray application reduces the amount of penetrant in the air.
2. If the test object has been immersed, drain the penetrant as long as possible to reduce carry over.
3. Rinse away the postemulsifiable penetrant before applying hydrophilic emulsifier. This penetrant will separate from the rinse water and may be skimmed off or otherwise separated. This step can allow the rinse water to be recycled.

**Figure 8.** Typical wastewater from fluorescent penetrant test rinse stage.

4. Apply emulsifier sparingly and allow to dwell the required time. Spray techniques require less emulsifier than dip techniques. Hydrophilic emulsifier can be applied using appropriate electrostatic spray equipment.
5. Rinse waters can be treated to separate out the emulsifier and penetrant by using special membrane filters. Activated charcoal can be used to remove the color, oil, and most of the emulsifier (wetting agent).
6. Filtration of rinse water will remove developer residues, if any.

Specific recommendations for waste disposal treatment and the cost of such a system depend on numerous factors, such as (1) the type of penetrant or emulsifier used, (2) whether application is by dip or spray, (3) the volume and type of work being processed, (4) the facilities and equipment in use, and (5) the particular restrictions of applicable city, county, state, or federal codes.

## TREATMENT FOR LIQUID PENETRANT EFFLUENTS

The actual effluent arising from a penetrant process generally consists of a dilute emulsion formed by direct emulsification of the penetrant with water, in the case of water washable penetrants. Similar dilute emulsions can be formed by the action of an auxiliary emulsifier or penetrant remover in the case of postemulsifiable penetrants. The auxiliary emulsifying agents may contain constituents broadly similar to those of the penetrants themselves, and the surfactant system may be either lipophilic or hydrophilic in nature. The effluent emulsion generally contains less than one percent by weight of nonaqueous matter and is usually fairly stable. Conventional treatment can be designed to break the emulsion and thus separate nonaqueous matter. It involves collecting and treating the effluent successively with strong electrolytes and flocculating agents in a series of vats or tanks.

The coagulated contaminants are separated by filtration and finally incinerated, leaving water of acceptable purity for discharge or reuse. This is essentially a batch process, relying for its efficiency on accurate pH control. It also requires time and space: time for the various additives to become fully effective and space to accommodate the plant, the size of which is determined by the volume of effluent requiring treatment in a given period of time. Where penetrant processes are used intensively and continuously, correspondingly large volumes of water are required for rinsing. Membrane filtration is one way of treating the effluent on a continuous basis and feeding back the purified water into the process. In effect, creating a closed circuit system that uses water economically.

## LIQUID PENETRANT WASTE IN SEWAGE AND STREAMS

When an inspection penetrant is rinsed from the test surface, it enters the rinse water as a fine, stable, oily emulsion. Typically, this emulsion is not considered to be toxic but does make the effluent turbid. It can leave an oil slick on the water's surface and even deplete some of the oxygen supply in the water. All of these results lower the value of the water. Sewage regulations in some localities allow plant effluent water to contain as much as 600 µg/g of such oils, whereas other regulations allow none at all. The rinsings from penetrant removal operations normally contain from 200 to 1000 µg/g oil and are usually not an acceptable waste.

### Measuring Concentration of Oily Contaminants in Wastewater

Oily contaminants can be detected either as hexane solubles or by chemical oxygen demand (COD). Extraction of rinsings by hexane yields their total nonvolatile oil content. The chemical oxygen demand test measures the concentration of organic contaminants (oil, surfactants, and so forth) by the amount of oxygen used in oxidizing them completely by a dichromate reflux technique. The results are expressed in milligram of oxygen per liter of test solution or in parts per million. One gram of a typical oily penetrant may consume about 2.5 to 3.0 g of oxygen in this manner. Thus, 1 L of rinsings containing 1 g of oily penetrant may show a chemical oxygen demand of 2500 to 3000 µg/L. More dilute rinsing may show a lower chemical oxygen demand. The chemical oxygen demand technique of monitoring pollution concentration can be related empirically to the biological oxygen demand. The biological oxygen demand test measures the oxygen used during a specified incubation period for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron.

### Avoidance of Environmental Water Contamination by Penetrants

Where local regulations allow some oil to be present in the plant effluent, the penetrant rinsings, combined with other nonoily waste streams from within the plant, may yield a total effluent within the acceptable range. In most cases, though, the situation is not this simple. Then something must be done with the penetrant removal operation to lower the oil content of the effluent.

Worldwide, many attempts have been made to manage penetrant removal without contaminating the environment. Some of these techniques use penetrant compositions that meet prevailing pollution control regulations. Other control techniques alter the process so that the wastes never leave the test area. Still other techniques treat the rinsings to concentrate the oily contaminants for easy disposal.

## COMPOSITIONS OF PENETRANT TEST MATERIALS TO AVOID POLLUTION

One approach to the pollution problem is to alter the penetrant's composition so that the rinsings are less objectionable. For instance, formulating a penetrant to be completely water soluble would avoid the formation of turbidity and oil slicks (Goff and Robinson 1998). Such rinsings might pass all tests, even though the rinse water would still be contaminated. However, complete solubility makes effluent treatment more difficult. Penetrants that are completely water soluble have been available since 1953.

## BIODEGRADABLE PENETRANTS TO REDUCE POLLUTION

Another way to alter a penetrant's composition is to make it biodegradable. Such a penetrant need not be water soluble, yet it can have the same removal properties as oily penetrants. The rinsings consist of the same sort of fine emulsion but with an important difference. Within a few days, the oily penetrant is almost completely decomposed by organisms in the water. The penetrant decomposition requires a large amount of free oxygen from the stream that contains the oily wastes. This behavior limits the locations where such a penetrant can be legally used.

It would be objectionable if penetrant effluents were emptied directly into natural bodies of water where the oxygen depletion could harm the environment. Oily waste processed in a sewage treatment plant in the presence of ample oxygen and a large population of hungry microorganisms will disappear without harmful effects. This will increase the workload of the sewage treatment plant and perhaps result in increased sewer charges. Biodegradable types of penetrant testing materials are commercially available where suitable treatment plants are nearby.

## DEVELOPMENTS IN PENETRANT WASTEWATER DISPOSAL

The increasing regulatory restrictions on water pollution brought greater scrutiny of penetrant test materials getting into wastewater (Robinson 1991). Tests were conducted to develop a better understanding of wastewater characteristics resulting from penetrant test materials introduced around 1990 (Holmgren and Plamoottil 1992). The focus was on two distinct formulation approaches: compositions based on petroleum distillates and those based on surface active agents (surfactants), more commonly referred to as biodegradable penetrant. The study investigated the treatability of wastewater generated in the normal course of fluorescent penetrant testing.

The popular theory at the time was that surfactant based penetrants, because they were oilfree, were more biodegradable than the traditional oil based formulations (Holmgren 1989), hence making them more drain disposable. However, the study came to the conclusion that the popular theory may not be true. This very limited study found that each of the studied factors important to a publicly owned treatment works — treatment time, inhibition, respiration rate, percent biological oxygen demand removal — is more favorable with an oil based penetrant than with a surfactant based penetrant. It further showed that even oil free penetrants test positively for oil when tested in the laboratory.

### Wastewater Disposal into Publicly Owned Treatment Works

The greatest challenge to penetrant users is knowing whether wastewater from penetrant processes can be sent out to publicly owned treatment works (Figure 9) through the sewer system (Hessinger and White 1998). The first step in finding the answer is to ask the operators of publicly owned treatment works if penetrant wastewater is acceptable for treatment at these facilities. Wastewater treatability depends on how microorganisms in wastewater treatment plants respond to the waste stream. Conducting tests on fluorescent dye penetrants using a respirometer simulated the treatment of penetrant test materials in the activated sludge system. Activated sludge consists of microorganisms cultured in a controlled environment where wastewater enters and clean water (effluent) exits. The microorganisms are fed oxygen, mixed, retained for a period of time, and then settled. Most waste treatment plants use the activated sludge treatment system to reduce the soluble (dissolved) organic strength waste before the wastewater is discharged to a river, stream, or lake.

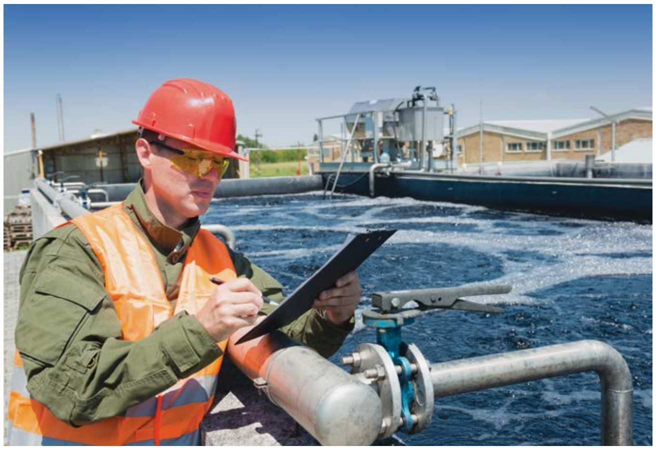

**Figure 9.** Wastewater treatment plant.

This reduction in organic strength is accomplished not only because the United States Environmental Protection Agency requires it, but also because the wastewater would quickly deplete the oxygen in the receiving stream. The publicly owned treatment works' objective is to remove organic and solid and soluble organic waste. Anything that threatens the achievement of this goal is a concern of the facility.

### Drain Disposability

Drain disposability is a term loosely used to denote the environmental friendliness of a particular waste when it is put in a sewer system. In the case of activated sludge systems, drain disposability would mean that the wastewater can be treated without hurting the microorganisms, within the capability of the air supply system (aeration), and within the treatment time (detention time). Drain disposability largely depends on the strength of the waste. Strength of waste is measured with the five-day biological oxygen demand test, which measures the depletion of oxygen from primarily microbiological metabolism (respiration). The higher the biological oxygen demand, the larger the tank capacity and air requirements. Penetrant test materials that result in low biological oxygen demand values are more likely to be treated within the detention time allotted by publicly owned treatment works to treat wastewater. In some tests, all oil based materials tested resulted in oxygen uptake rates below normal requirements, whereas most surfactant based samples tested exceeded the maximum allowable range. In summary, all penetrant products exerted a certain amount of organic strength measured as biological oxygen demand and thus may require pretreatment before disposal to publicly owned treatment works.

## POST-TREATMENT OF LIQUID PENETRANT RINSINGS FOR POLLUTION CONTROL

Several penetrant waste purification processes result in water pure enough to reuse in the rinsing step. Most of these processes rely on post-treatment of the rinsings. Post-treatment processes for penetrant rinsings can be placed into four groups: (1) chemical destruction of the emulsion; (2) molecular filtration, such as reverse osmosis; (3) absorption of the emulsion onto a suitable substrate; and (4) a remover that forms an unstable emulsion that separates easily. Each of these techniques of pollution control is described in detail below.

## Chemical Destruction of Liquid Penetrant Removed with Water

Surface active agents that allow penetrant removal by water also stabilize the resulting emulsion. These surfactants can be destroyed by oxidizing agents. One of the most effective oxidizing agents is potassium permanganate. With the surface active agent destroyed, the fine emulsion can separate. The oily constituent is absorbed onto the manganese dioxide floc (loose clump of fine particles) that forms and settles out when the permanganate reacts. This oxidation is slow. If permanganate is added to the collected rinsings in an amount equal to that of the penetrant and the mixture is not heated, it takes about 2 h to break the emulsion. At this point, a small excess of ferrous chloride must be added to the collected rinsings to decompose any unreacted permanganate. The resulting ferric oxide floc helps absorb more penetrant.

The penetrant floc mixture can be easily separated by filtration or decantation, but the technique is slow. Pound for pound, permanganate is more expensive than penetrant. Finally, the chemical additions must be painstakingly balanced so that something more deleterious than the original penetrant does not go down the sewer unneutralized.

## Reverse Osmosis for Removal of Waste

In reverse osmosis, the impure water is forced through an osmotic membrane under pressures up to 2.80 MPa (400 $\text{lb}_f/\text{in.}^2$). In a single stage, from half to three fourths of the water passes through the membrane. The contaminants that cannot pass through the membrane are concentrated in the remaining wastewater. Although such equipment is primarily used to separate pure water from mineral laden water, it also works well with water washable penetrants that are surfactant based.

Figure 10 shows schematically how a membrane separator works and includes a simple flow diagram. This is a soluble oil waste treatment system in which the separation is nearly quantitative. Beginning with municipal water laden with 2000 µg/g of emulsified penetrant (chemical oxygen demand about 4000 µg/g), it is possible to salvage 65 percent of the water. This recovered water contains less than 10 µg/g impurities, largely a water soluble emulsifying agent, and has a chemical oxygen demand of 60 µg/g. The wastewater is about

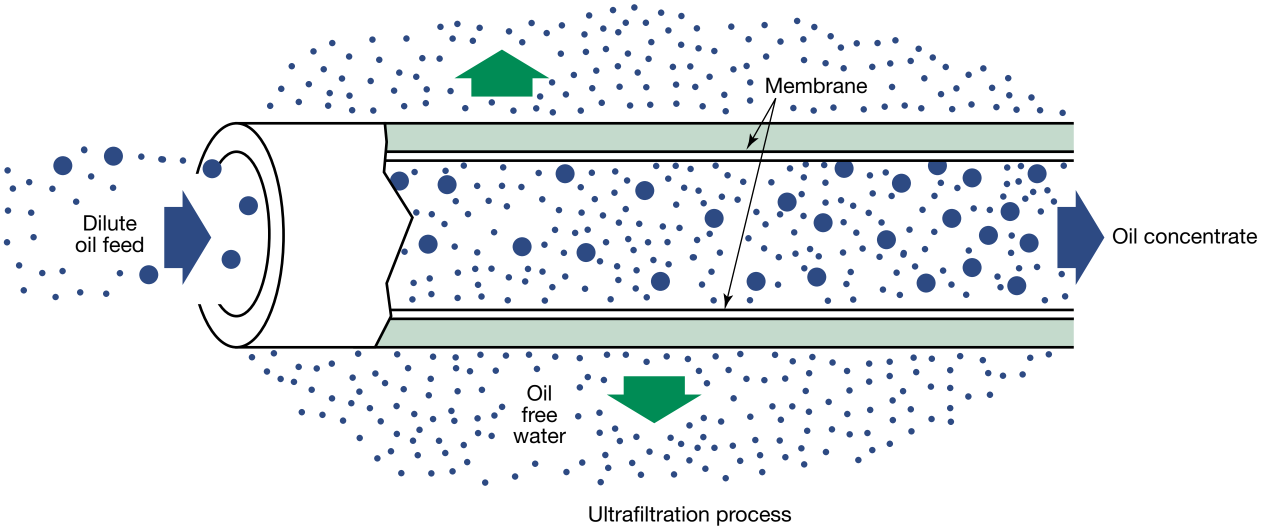

**Figure 10.** Schematic cross section drawing of permeator showing membrane used to pass oil free water radially outward and retain oily contaminants in tubular enclosure.

35 percent of the total. This wastewater contains over 90 percent of the mineral content of the original sample and all but a trace of the original.

Its chemical oxygen demand is around 18 000 µg/g. The reverse osmosis process concentrates the oily contaminant into 35 percent of the original wastewater, which is still too bulky for disposal. However, this concentrated waste can be forced through another osmotic membrane to salvage more pure water and shrink the volume of the wastewater.

## Performance of Osmotic Liquid Penetrant Removal System

In one series of tests with the permeator of Figure 10, penetrant rinsings were subjected to three stages of purification. Overall, about 94 percent of the rinsings was salvaged as pure water. The efficiency of the separation, as well as the rate of output, dropped as more concentrated water was fed to the permeator. The purified water from the first stage separation had a chemical oxygen demand of 60 µg/g. By the third stage, enough impurities were passing to raise the chemical oxygen demand to 110 µg/g. Table 2 shows the chemical oxygen demand values for the initial rinsings along with the values for reject and product water for each of the three successive stages.

**Table 2. Effect of successive rinses on waste water purity.**

| | Chemical Oxygen Demand (µg/g) | |
|---|---|---|
| **Stage** | **Waste** | **Product** |
| Initial | 4000 | |
| 1 | 18 000 | 50 |
| 2 | 32 000 | 75 |
| 3 | 66 000 | 110 |

This does not represent much contamination, but there is another limit on the degree of waste concentration that can be obtained. The mineral content of the wastewater also increases at each stage. Eventually it exceeds the solubility and precipitates out to form scale on the membrane, which stops further action. The scale can be removed, but only by shutting down the permeator and flushing it out with scale dissolving chemicals.

The permeator can treat from 6 to 10 L (1.5 to 2.5 gal) of water per minute, depending on the impurity level, pressure, and separation ratio. Unfortunately, it is damaged by traces of chlorine and oil in water. In addition, it still requires disposal of a significant volume of waste materials.

## Filtration

There are a variety of membrane materials and configurations. Membranes are made from polymers, stainless steel, and ceramics. Membrane selection is based on the size of the particles to be separated and the chemistry of the effluent stream as it relates to the chemistry of the membrane.

The standard membrane delivery system is a “dead end” filtration, so called because the feed fluid flow is directed at a right angle to the membrane without any attempt to control the thickness of the concentrate boundary layer at the filter’s surface. Rapid buildup of retained solids to the membrane surface and continuous buildup of materials severely limit the flow and separation of the effluent. The most widely used dead end filters are cartridge filters. Generally, cartridge filters are limited to feed streams of low viscosity and solid content. Once the filter flow rate has dropped off to an unaccepted level, the cartridge filter must be discarded.

To achieve relief from the accumulation of rejected effluent materials and the increasing pressure drop, filtration systems have been developed to flow the effluent stream parallel to the membrane’s surface in filtration called cross flow or tangential. In tangential flow filtration, the effluent feed is pumped at a high velocity in an attempt to shear away the concentration polarization layer and minimize its effect on the separation. Turbulent flow designs operate at high shear rates and achieve higher filtration efficiencies.

Conventional tangential flow filtration configurations include plate and frame, spiral wound (stacks of membrane sheets with turbulent promoting screens between layers), hollow fibers (bundles of filament membranes), and tubular designs (membrane tubes). The spiral wound configuration is easily serviced and is the most energy efficient tangential flow filter design. It is used in the food, chemical, and environmental industries for ultrafiltration and reverse osmosis applications.

## REUSE OF SEPARATED PURE WATER

The separated pure water can be drained into the sewer or it can be reused in the penetrant removal process. In the permeator tests described in Table 2 and Figure 10, the small amount of impurities that passed through the first stage did not increase after six cycles of penetrant reuse and contamination. Table 3 shows the chemical oxygen demand values of the product water after six cycles of reuse. Different penetrants should be checked for compatibility and processing characteristics.

**Table 3. Purity of separated water after six cycles of reuse.**

| Cycle Number | Chemical Oxygen Demand (μg/g) |
|---|---|
| 1 | 60.8 |
| 2 | 57.6 |
| 3 | 57.2 |
| 4 | 64.0 |
| 5 | 48.0 |
| 6 | 64.0 |

## LIQUID PENETRANT REMOVAL BY ADSORPTION

Studies have shown that the clarification of effluent containing penetrant waste also can be accomplished by the adsorption technique. It is essentially based on the affinity of certain absorbent particles toward typical ingredients of penetrants. In practice, the oil contaminant is extracted by stirring vigorously 7 kg (15 lb) of absorbent into 10 000 L (2600 gal) of wastewater containing about 0.06 percent penetrant. A solution of a flocculating agent is then added and the treated batch can be separated by any of the following techniques: (1) stratification, (2) filtration, or (3) centrifugation.

1. Flocculated solid particles settle loosely at the bottom of the tank, allowing the purified water to be decanted off. The bottom layer (about 20 percent of the original volume) will then have to undergo further processing such as evaporation or filtration to reduce it to a more easily disposable, compact, wet solid waste.
2. The filtration of the treated effluent batch can be accomplished by using a precoated horizontal plate filter. The end products of this technique are purified water (oil content is about 25 μg/g) and a wet solid cake that has only slightly more bulk than the removed penetrant itself.
3 In Table 2 and Figure 10, successful extraction of solid particles of clay from the treated batch of effluent was also achieved by means of a simple cream separator. However, a continuous separation would require special equipment that, although industrially available, is much more expensive than an ordinary centrifuge.

## WATER IMMISCIBLE SOLVENT REMOVERS

Another approach to the effluent problem is the removal of non–water washable penetrants by aqueous dispersions of volatile, water immiscible solvents. This results in an effluent whose typical composition is as follows: (1) 99 percent water, (2) 0.98 percent solvent, and (3) 0.02 percent penetrant.

The mutual compatibility of the penetrant and solvent remover and their combined immiscibility in water predetermines an easy separation of water by centrifugation or gravity stratification in a holding tank. Apart from exhibiting a slight bluish fluorescence, the recovered water is sufficiently uncontaminated (oil content less than 100 μg/g) to be disposed of as a regular aqueous waste. The density of the removing solvent must be either lighter or heavier than water in order for it to be separated by centrifugation or gravity. The considerably smaller volume of the remaining remover penetrant mixture can then be distilled to recover the volatile solvent, leaving behind proportionally minute quantities of somewhat contaminated penetrant. Or it can be skimmed off and collected for disposal as oily waste. The solvent fraction will have mostly penetrant oils and traces of water and can be incinerated or used as a fuel blend.

It follows that the solvent removal of penetrants represents a foundation for a closed circuit system that eliminates effluent entirely by reusing water and the remover, and by collecting the penetrant waste in concentrated form.

## LIQUID PENETRANT WASTE PURIFICATION WITH ACTIVATED CARBON

The technique of filtration through activated carbon and other filter media for decolorizing is a well known and established technique. It is usually carried out by making a slurry of carbon in the liquid from which the color is to be removed, and by heating it, if possible. After a suitable reaction time, which may vary from a few minutes to several hours depending on the nature of the coloring matter and its concentration, the carbon is separated by filtration. Where large amounts of color have to be removed, it is generally necessary to repeat the process several times. It is a batch process limited by time and space constraints.

However, it has been found that it is possible to achieve the same end by allowing the effluent to flow continuously through a bed of activated carbon and other filter media. Two major factors determine the success of this technique. First, the type and particle size of carbon and filter medium are specific for a particular emulsion and apparently depend on its hydrophilic lipophilic balance (HLB). Secondly, the ratio of the rate of flow of effluent to the volume of carbon is highly critical in determining the rate at which color is removed. This in turn varies with the amount of contamination present in the carbon and filter medium, so that the rate of removal of color reduces as the degree of contamination of the filter media increases.

### Waste Purification by Carbon Filtration

Waste purification is not simply a matter of removing the organic dyes by a process of adsorption but rather a combination of this and filtration. This is confirmed by a combination of activated carbon and filter aid, such as pearlite or diatomaceous earth, being more effective than either one of these on its own. If only carbon is used, the oily matter in the effluent quite soon forms an oily coating on the carbon particles that effectively prevents their functioning as adsorbents for the dye. If only pearlite or diatomaceous earth is used, only a filtration effect is attained, with little or no adsorption of dye taking place.

Experimental work has shown that a sandwich arrangement, whereby the effluent flows first through the filter aid and subsequently through the activated carbon, practically doubles filter life, that is, its capacity to function before reaching actual or apparent saturation.

### Recycling of Purified Water after Carbon Filtration

Carbon filtration, with the correct grades of carbon and filter aid, will produce clean water that may be discharged to waste. The only contaminants that may still be present in the water are the strongly hydrophilic elements of the surfactant system of the penetrant or emulsifier. If the water is to be discharged to waste, it is important that only surfactants that can be readily and substantially broken down by biological organisms like biodegradable detergents be used in the penetrant or emulsifier formulation.

In the present state of knowledge, the choice of suitable nonionics that also meet the other required criteria is limited. However, a postemulsified penetrant process in which a hydrophilic penetrant remover is used presents a further problem if the rinse water is recirculated. Under such conditions the concentration of penetrant remover in the rinse water gradually builds up to a level where it causes excessive foaming. This foam is deposited and dries on the test objects being processed, staining them and thus hindering inspection.

## ADSORPTION OF SURFACTANTS ONTO CARBON

Certain types of carbon are capable of removing surfactants. However, it was discovered that types of carbon that remove surfactants have no decolorizing properties with respect to the type of dye generally used in fluorescent penetrants. Therefore, one has to adopt a sandwich arrangement of the two types of carbon, separated by a perforated plate, set up so that the effluent water passes first through the layer of decolorizing carbon and thereafter through the layer of carbon that removes the surfactants. It was also found that if the order of the two carbon types is reversed or if they are intimately mixed or blended, the desired effect is not produced. Reverting, therefore, to what has been said previously about filter aids, a three layered sandwich consisting of one layer of filter aid and two layers of carbon is required for some applications. Disposal of the saturated carbon is best carried out by incineration, which destroys the adsorbed organic matter. If the quantities justify it, regeneration of the carbon may be economically attractive.

### Carbon Filters with Hydrophilic Emulsifiers

In view of the relatively high price of activated carbon, the cost of the carbon filter process is significant and ways of improving the economics have therefore been sought. One technique that shows worthwhile savings is a postemulsifiable hydrophobic penetrant in conjunction with a hydrophilic penetrant remover. In such a system, a preliminary water rinse is carried out before immersion in the penetrant remover. This rinse removes the bulk of the excess penetrant by mechanical action without forming an emulsion. Then, this penetrant may be recovered from the water by a centrifuge or coalescer. Both achieve almost 100 percent separation, producing clean water for reuse or discharge and actually recovering usable penetrant. In one particular installation, about 4 L (1 gal) of penetrants (representing about 65 percent by volume of the total dragout) are recovered daily. This process, which may be carried out continuously, achieves a double effect. It economizes in expensive penetrant and, by ensuring that far less penetrant is carried over to the second rinse, reduces the amount of penetrant to be removed by the carbon filter, thus increasing its service life and reducing the operating costs of the process.

# Part 4

# Recycling of Water Effluent and Postemulsifiable Liquid Penetrant

## HYDROPHILIC PROCESS FOR RECOVERY OF LIQUID PENETRANT

The prewash or prerinse concept, which uses a postemulsifiable penetrant and a hydrophilic emulsifier, is known as the hydrophilic postemulsification penetrant process. This process permits recycling of the wash water. The technique calls for removing the bulk of the postemulsifiable penetrant from the surface with a plain water wash before treatment with an emulsifier. Properly formulated postemulsifiable penetrant used in the prewash mode will yield nonemulsified effluent that separates by gravity when agitation ceases. The penetrant should float for easy removal by skimming and for possible reuse if carefully collected. The water that remains on the bottom of the collection tank may also be reused in the prerinse step. Further processing may be required if separation is incomplete as evidenced by discoloration or organic contamination. The prewash concept can be used in closed loop penetrant processes where wastewater and unexpended penetrants are recycled through the system rather than expelled into the environment.

## PREWASHING FOR RECOVERY OF LIQUID PENETRANT

Another approach to control of penetrant waste pollution uses a non–water washable penetrant and gravity separation. This system permits recycling of the penetrant as well as the rinse water. The technique calls for removing the bulk of the non–water washable penetrant from the surface with a plain water wash before treatment with an emulsifier. Therefore, the principal effluent of prewashing is a nonemulsified mixture of penetrant oil and water; the mixture quickly and completely separates by gravity. The penetrant is skimmed off. The water is drawn from the holding tank and recirculated. The prewash concept can be useful in closed loop penetrant processes where wastewater and unexpended penetrants are recycled through the system rather than expelled into the environment.

Data have shown that higher penetrant system performance and greater reliability are possible with prewashing (Birley, 1969). Emulsifier contact time is less critical in this system than in lipophilic postemulsification.

### Potential Advantages of Prewashing

The prewash system addresses two contemporary exigencies: conservation of petroleum derived products and water pollution control. It also results in lower material costs, and pollution abatement expenses will be lower because the primary wash water is recycled without treatment and the second wash water is recycled with only minimal treatment. Recycling is accomplished with a penetrant system believed to provide higher levels of performance and reliability than water washable techniques.