OxyChem®
Basic Chemicals

Chlorinated Organics
Liquid Dryer Information

Introduction
Liquid dryers can be used to remove small amounts of dissolved water from solvents. This removal becomes necessary because the presence of water in chlorinated solvents can cause decomposition and corrosion. A water separator usually precedes a liquid dryer for removal of any phased (non-dissolved) water in the solvent stream because the operation of a liquid dryer for this purpose is not economical. Following the liquid drier, about 5-50 ppm (parts per million) water will typically still remain in the solvent.

To accomplish the removal of water using a liquid dryer, the liquid solvent is passed through a freshly regenerated column packed with desiccant material which selectively holds the water and allows the solvent to exit the dryer bed. Each desiccant has a characteristic range of water capacities and must be removed from service at regular intervals to be regenerated or replaced. Continuous operation may be achieved by using dual dryer columns with one column on the solvent stream while the other is being regenerated.

Desiccants
Several desiccants are commercially available for drying chlorinated solvents, including ion exchange resins (Dowex® HCR-W2, Na form), molecular sieves, anhydrous calcium sulfate (Drierite®), silica gel, activated alumina, and anhydrous calcium chloride (Peladow®) (see Table 1). The Dowex® resin is generally well suited for solvent drying, as it has the advantages of high sorption capacity and low temperature regeneration. Molecular sieves have high sorption capacity and are unique among the common desiccants in their capability to remove water from high temperature streams. Drierite® is a suitable desiccant and has a reasonable price, but it requires filters after the dryer bed to trap extremely small fines that can escape from the bed and become deposited in pipes, pumps, and tanks. Though recommended for use in air drying, silica gel can sometimes be used to dry liquid solvents; however, the regeneration temperatures necessary to achieve good water removal tend to shorten the effective life of the desiccant by damaging its internal structure. Activated alumina is best suited for extremely low temperature liquid streams, but it also tends to have a short effective life for the same reason as silica gel. The best application for calcium chloride is as a disposable desiccant used on a short-term basis. Calcium chloride is not regenerable but is relatively inexpensive and has a high sorption capacity.

Dryer Design
Although liquid dryer units are commercially available from a few manufacturers, it is probably wise to become familiar with some general design guidelines before purchasing any equipment. Common knowledge of suitable design parameters for liquid dryers

OXY
Wichita Technical Service Department
6200 South Ridge Road, Wichita, KS 67215
Tel: 800-733-1165 ext. 1
OxyChem_Tech_Service@oxy.com

is rather limited; in some cases, it may be advisable to design a drier rather than use a manufacturers' design. Table 2 is a compilation of some suggested design guidelines extracted from commercial information on desiccant dryers. This information is primarily concerned with the sizing of dryer units.

In addition to these design guidelines, there are additional considerations in liquid dryer design. Most desiccant dryers are designed to remove about 96-97.5% of the water from the inlet solvent stream. Since the solubility of water in chlorinated solvents differs substantially (see Table 3), the amount of desiccant needed to dry a particular solvent will also differ, and this should be taken into consideration when sizing the dryer. In addition, the volume of desiccant required for a drying operation may be reduced by using slower flowrates or by having greater height-to-diameter ratios (H/D) in the drying column. However, high H/D ratios increase the pressure drop across the bed, which will usually run up to about 1 psi per foot of bed height. Adsorbent-type desiccants (ion exchange resins, molecular sieves, silica gel, and activated alumina) will expand slightly when they adsorb water and may arrive from the manufacturer up to 100% larger than their activated size. The drying capacity of the desiccant is extremely important in dryer design for proper sizing of the dryer bed. In addition, sometimes it is wise to over-design the dryer bed by about 20-25% to ensure that sufficient drying capacity will be available throughout the entire drying cycle. An example of a basic dryer design is shown in Appendix A.

Regeneration

Since any desiccant has a finite limit to the amount of water it will hold, it must either be replaced or regenerated periodically in order to provide continued removal of water from the solvent stream. The regeneration, or reactivation, of a liquid dryer is accomplished by first draining the dryer of liquid solvent, and then heating the desiccant to drive off the trapped water. This is usually achieved by passing a hot, dry air stream through the bed. The temperature of the hot air purge necessary to adequately regenerate a dryer bed depends on the type of desiccant used; these values appear in Table 1. The apparatus commonly used to provide the hot air stream is called a regeneration unit and consists simply of a blower and a heating chamber with electric elements for the heat source. The length of the regeneration cycle will depend upon the particular system and desiccant used but is usually approximately 8 hours. Regeneration of the desiccant can also be achieved by removing it from the dryer and baking it in an oven, but this is seldom desirable due to the substantial amount of time required for handling the desiccant. Steam is not recommended as a heat source for regeneration.

For dual bed drying columns, the columns must be designed such that each drier has sufficient capacity to operate effectively throughout the entire period required to regenerate the other dryer. The dryer's operating instructions should include a guide as to when regeneration is needed. If this is not the case, testing the solvent for water content before and after it passes through the dryer should indicate when the dryer needs to be regenerated. Testing for water content can be done by either the Karl Fischer Method (ASTM D 3401) or by the Cloud Point Method for all chlorinated solvents. Certainly, if any free water is observed in solvent that has passed through a dryer, regeneration is long overdue.

Equipment Suppliers
The following is a partial list of manufacturers of liquid drying equipment:

Lectrodryer LLC
PO Box 2500
Richmond, KY 40476-2602
859-624-2091
www.lectrodryer.com

Parts Cleaning Technologies
12886 Eaton Avenue
Detroit, MI 48227-3949
313-491-4550
www.partscleaning.net

Regeneration Equipment

Industrial Electric Heating Supply
5110 NW 86th Terrace
Kansas City, MO 64154
816-587-2161

Further Information
More detailed information is available on request through the OxyChem Technical Services Department, call or write to:

OxyChem Technical Service Department
6200 S. Ridge Rd.
Wichita, Kansas 67215
800-733-1165 option #1
OxyChem_Tech_Service@oxy.com

TABLE 1
Relative Properties of Various Desiccants Used in Chlorinated Solvent Drying

Desiccant	Approximate Capacity lb. Water Sorbed Per lb. Desiccant	Regeneration Temperature Recommended °F	Maximum Allowable Regeneration Temperature °F	Maximum Allowable Solvent Stream Temperature °F	Bulk Dry Density lb./ft^3	Water Removed Per Bed Volume Estimate lb. H_2O/ft^3	Comments*
Ion Exchange Resins (Dowex® HCR-W2, Na form)	10-15%	275-300	325	<200	57	5.7-8.5	Expensive, but has excellent sorption capacity and low heat requirement for regeneration.
Molecular Sieves	5-15%	400-600	950	<600	46	2.3-6.9	Relatively expensive, but has ability to dry high temperature liquid streams.
Anhydrous Calcium Sulfate (Drierite®)	5-6%	375-450	575	<200	60	3.0-3.6	Reasonable price. Fine particles can wash out of bed and cause problems if not filtered out. Do not use Du-Cal type.
Silica Gel	4-6%	350-500	500	<125	45	1.8-2.7	Suggested use is for gas drying rather than liquid drying. Sometimes has short life due to tendency to coke and spall during regeneration.
Activated Alumina	5-8%	350-600	600	<125	55	2.7-4.4	Sometimes has short life due to tendency to coke and spall during regeneration. Has ability to dry unusually low temperature liquid streams.
Anhydrous Calcium Chloride	5-55%	Not Regenerable	Not Regenerable	<200	64	3.2-6.4	Not regenerable. Relatively inexpensive way to remove water on a short-term basis. Do not use to dry 1,1,1-trichloroethane.

* Many desiccants are sold in moisture-swollen form, meaning that the actual price of activated desiccant is much higher (up to 100% higher). More specific information may be obtained from manufacturers on request.

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TABLE 2 **Guidelines for Design Parameters of Desiccant Dryers**		
Parameter	**Guidelines Suggested**	
	Range	**Specific**
Bed height to diameter ratio (H/D)	1.5/1 – 4/1	3/1
Flow rate through bed per cross-section	5-10 gpm/ft^2	7.5 pgm/ft^2
Residence time of solvent in bed	3-10 min	4.5 min
Superficial velocity of solvent through bed	0.7-1.3 ft/min	1 ft/min
Maximum height of bed	----------	7 ft.

TABLE 3 **Solubility* of Water in Chlorinated Solvents @ 25°C**	
Solvent	**Solubility**
Carbon Tetrachloride	125 ppm
Chloroform	850 ppm
Ethylene Dichloride	1500 ppm
Methylene Chloride	1700 ppm
Perchloroethylene	105 ppm

* Values are only approximating, because water solubility in liquid solvents depends partly on the particular stabilizer system used.

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OxyChem_Tech_Service@oxy.com

TABLE 4 Density of Chlorinated Solvents @ 25 C	
Liquid Chlorinated Solvent	**Density (lbs/ft^3)**
Carbon Tetrachloride	98.39
Chloroform	91.96
Ethylene Dichloride	78.03
Methylene Chloride	86.09
Perchloroethylene	100.9

APPENDIX A: Desiccant Dryer Design Example

GIVEN: Methylene Chloride at a flow rate of 10 gal/min and temperature of 25°C is saturated with water, but free water has already been removed in a water separator. Approximately 96% of the water is to be removed in a liquid dryer. It has been found that it will take about 8 hours to fully regenerate an individual dryer bed.

FIND: An acceptable basic design for the continuous removal of water from the liquid solvent stream using Dowex® resin HCR-W2, Na form.

SOLUTION: **Liquid solvent flow rate = 10 gal/min**

Desiccant capacity = 10%
Bulk dry density of desiccant = 57 lb./ft^3
Inlet liquid solvent contains 1700 ppm water
Drying period before regeneration = 8 hours
Use H/D = 3/1 as a design guideline
Dual dryer columns needed for continuous operation
Use over-design factor of 1.25

Amount of water to be removed:

10 gal. wet solvent	1700 lb. Water	86.09 lb. solvent	1 ft^3	8 hrs.	60 min	= 93.9 lb. Water
Min.	10^6 lb. Wet solvent	ft^3	7.48 gal	cycle	hr.	cycle

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Amount of desiccant needed per bed:

$$\frac{93.9 \text{ lb. Water}}{} \times \frac{100 \text{ lb. Desiccant}}{10 \text{ lb. Water}} \times \frac{1.25}{} = 1173 \text{ lb. Desiccant per bed}$$

Volume of dryer bed:

$$\frac{1173 \text{ lb. Desiccant}}{} \times \frac{\text{Ft}^3 \text{ desiccant}}{57 \text{ lb. Desiccant}} = 20.58 \text{ ft}^3 \text{ bed volume}$$

Cylindrical bed volume:

$$V = \frac{(3.14)D^2H}{4} = 20.58 \text{ ft}^3 \qquad H = 3D$$

$$D^3 = \frac{(20.58)(4)}{(3.14)(3)}$$

$$D^3 = 8.74 \text{ ft}^3 \rightarrow D = 2.06 \text{ ft.} \qquad H = 6.18 \text{ ft.}$$

Residence time of solvent in bed:

$$\frac{20.58 \text{ ft}^3}{10 \text{ gal/min}} \times \frac{7.48 \text{ gal.}}{\text{ft}^3} = 15.39 \text{ minutes}$$

Flow rate of solvent per cross-section of bed:

$$\frac{10 \text{ gal.}}{\text{Min}} \times \frac{4}{3.14} \times \frac{}{(2.05 \text{ ft})^2} = 6.21 \text{ gpm/ft}^2$$

Superficial velocity of solvent:

$$\frac{6.21 \text{ gal}}{\text{min ft}^2} \times \frac{\text{ft}^3}{7.48 \text{ gal}} = 0.83 \text{ ft/min}$$

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