Dehydration with Ion Exchange (Dessicant)

(Extraction from the Diaion Manuals pages 354 to 360)

2. Dehydration/ Drying with IERs

(1) Outline

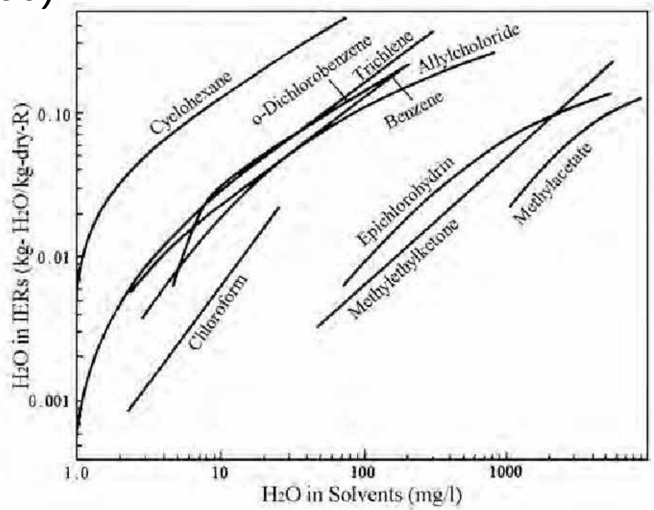
Silica gel, activated alumina and molecular sieves are traditionally used to dehydrate organic solvents and gases ultimately. IERs can also be used as superior dehydrating agents.

It has been empirically well-known that IERs become hygroscopic when they are dried. They can absorb selectively not only water but also polar solvents. Polar solvents can be removed from non-polar solvents selectively by using this IERs' property. Water is, in particular, easily absorbed by IERs. Thus, non-polar solvents and gases can be dehydrated by dried IERs effectively. This dewatering function of IERs is strong since it is caused by hydration of their functional groups with counter ions. The IERs that have absorbed water can be easily regenerated with hot air of low temperatures; ca. 120 °C.

(2) Mechanism Power of Dehydration

DIAION® SK1B, gel-type CER, in Na-form is used as a dehydration agent. The fixed ion, $-SO_3^-$ and the counter ion, Na^+, of this IER are easy to solvate polar solvents, and thus the IER absorbs polar solvents with its own swelling. On the contrary, it rarely absorbs non-polar solvent. Water, strong polar solvent, can be eliminated from non-polar solvents. Macro pores have affinity with non-polar solvents and thus can be used as dehydration agents, but they cause a large loss of the solvents. Thus, gel-type IERs are suitable for dehydration.

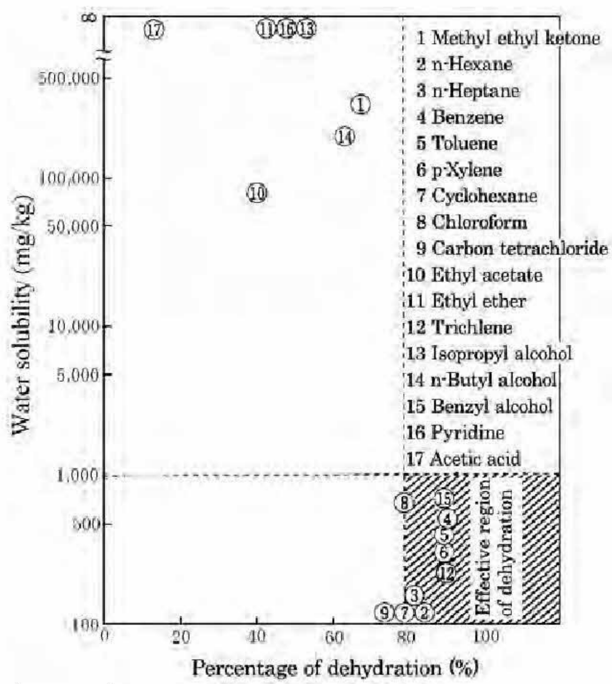
Fig.X-2-1 demonstrates the absorption equilibrium curves, the correlation curves between the absorbed water by IERs and the water in solvents in equilibrium, of various kinds of non-polar solvents. The difficulty of dehydration from different solvents can be compared based on these data. The curves of epichlorohydrin, methylethylketone and methylacetate shift from the other solvents in the right direction, and thus they show that these solvents have strong water absorbing power and poor dewaterability.



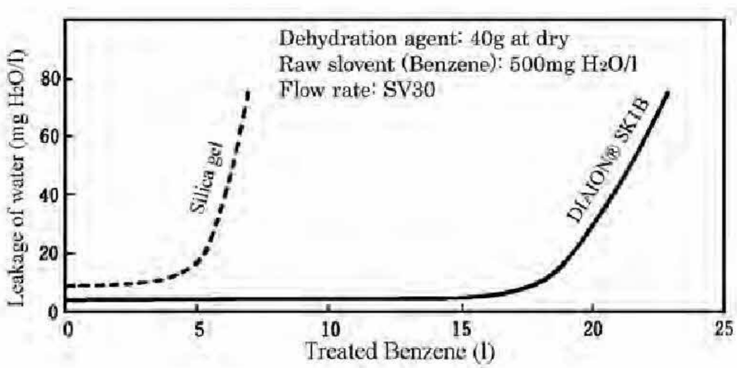
[Fig.X-2-1] Dehydration of various solvents with DIAION® SK1B
Absorption Equilibrium in Batch operations

These data suggest us that solvents with strong polarity and/or with high water solubility are generally unsuitable for dehydration by IERs. IERs are, in other words, very effective to remove extremely little amount of water from solvents with low water solubility. *Please see Fig.X-2-2.*

The dehydration by IERs is a kind of chemical hydration reactions. Its dewaterability is strong enough, similarly as concentrated sulfuric acid, to remove the residual water ultimately. As the actual dehydration operations, the column method is superior to the batch method practically and effectively. Figures X-2-3 through X-2-5 summarize the dehydration results of benzene, cyclohexane and air with IERs. The solvents dehydrated by IERs should be handled with the possible care to prevent, because they have very strong water absorbing power: e.g. not to contact with normal air, not to use laboratory equipments of rubber or vinyl chloride. Fig.X-2-6 illustrates a dehydration experiment arrangement in laboratories.

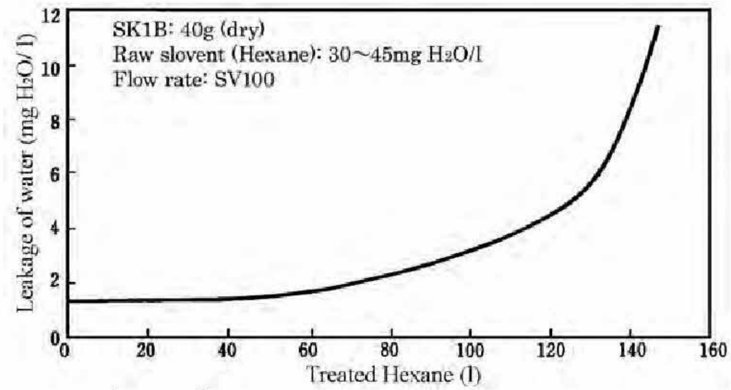


[Fig.X-2-2] Dehydration of Organic solvents with DIAION® SK1B
– Dehydration rate vs. Solubility of water –

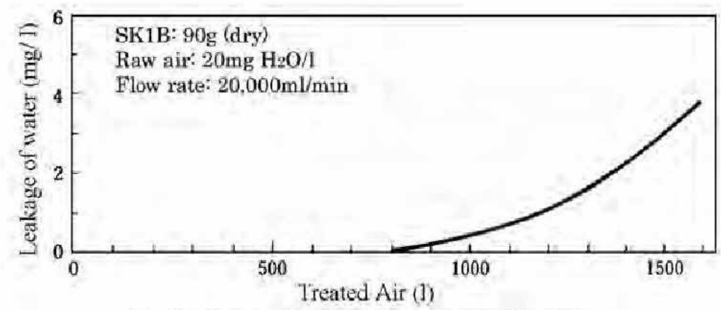


[Fig.X-2-3] Dehydration of Benzene with DIAION® SK1B

The water amount that IERs can remove from organic solvents depends on kind of the objective solvent, but is considered to be inversely proportional to the saturated water contents of solvents. Thus, the capacity can be estimated in Fig.X-2-7. IERs are estimated to dehydrate solvents the volume of which is two thousand times as large as IERs', if the water solubility of the solvent is 100 mg/L.



[Fig.X-2-4] Dehydration of Hexane with DIAION® SK1B

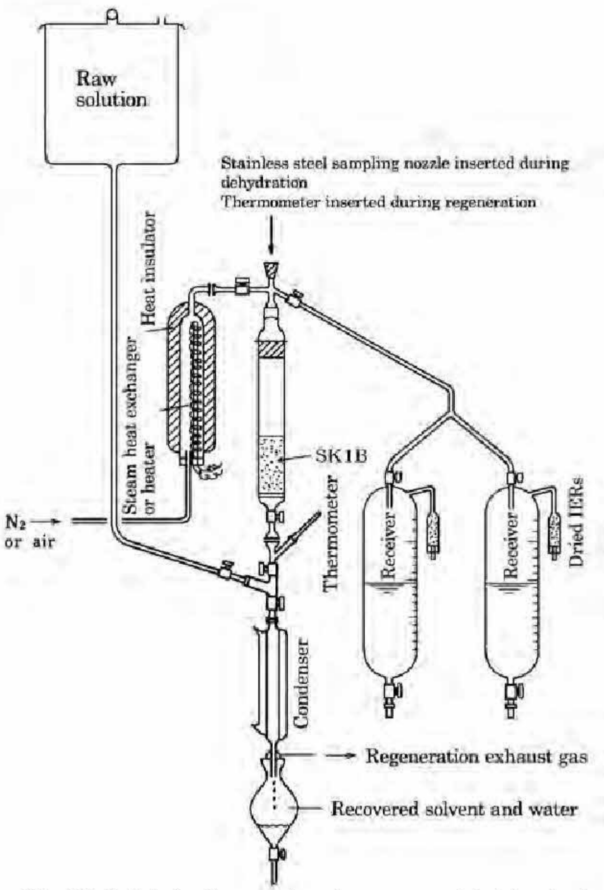


[Fig.X-2-5] Dehydration of Air with DIAION® SK1B

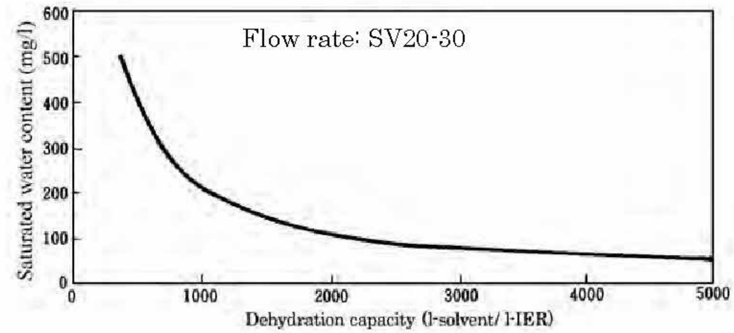
(3) Operation of Dehydration

There are two methods to dehydrate liquids or gases: one is the batch method in which dried dehydration agents are poured in the objective solvent and stand still or with stirring, and the other is the column method with continuous feed of the objective solvent through the dehydration agents. The column method is superior in effectiveness and in industrial use. A flowsheet of a standard actual plant is demonstrated in Fig.X-2-8.

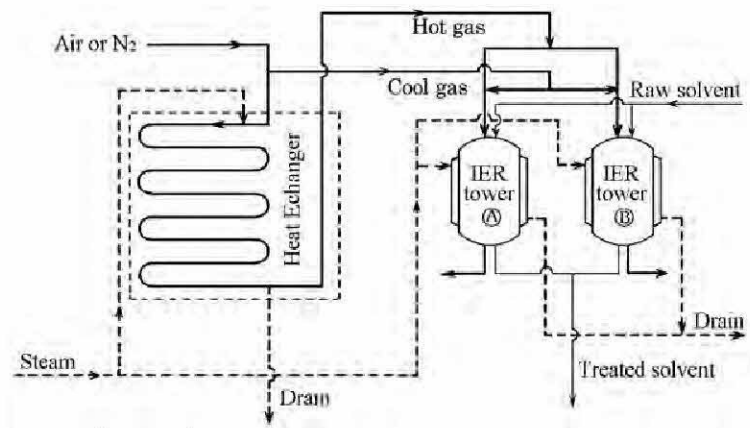
Dehydration agents are regenerated/ dried by downward feeding of hot air, 100 ~130 °C, or inert gas in towers. The temperature of outlet gas can be used as a monitor to know the end-point: it continues to be low due to the latent heat of vaporization and increases rapidly at the end-point. *Please see Fig.X-2-9.*



[Fig.X-2-6] Dehydration experiment arrangement in laboratories.

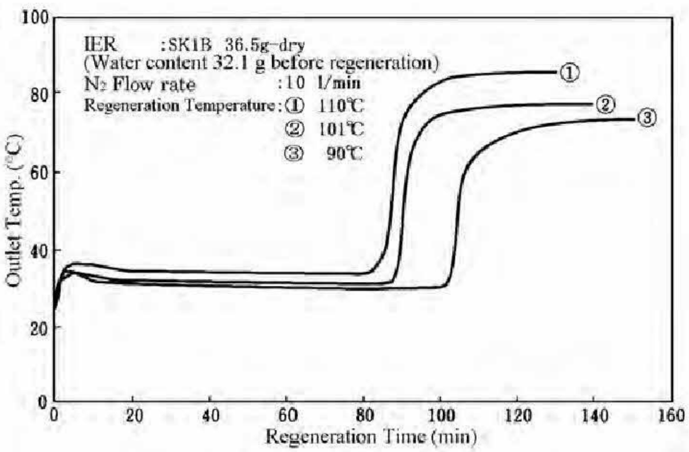


[Fig.X-2-7] Saturated water contents and Dehydration capacity of DIAION® SK1B



[Fig.X-2-8] Flowsheet of Dehydration Plant by DIAION® SK1B

The regenerated dehydration agents are cooled down by dehumidified gases, cooling jacket or both of them, and then reused for the next dehydration operation. The advantage of IERs as dehydration agents is that they can be regenerated by various methods and at lower temperatures, 100 ~130°C, than other dehydration agents, e.g. silica gel, 150 ~200°C, activated alumina, 200 ~300°C, and molecular sieves, 200 ~500°C. They have, on the contrary, disadvantages that the objective solvents are limited low polar ones and that the volume of IERs increases in accordance with absorbing water. Chloroform, benzene, toluene, xylene, trichlene, n-heptane, n-hexane, cyclohexane and carbon tetrachloride can be dehydrated effectively with IERs.



[Fig.X-2-9] Regeneration of IERs