NEW METHOD FOR AIR-FREE COLLECTION OF ULTRA DRY and DE-OXYGENATED SOLVENTS

INTRODUCTION

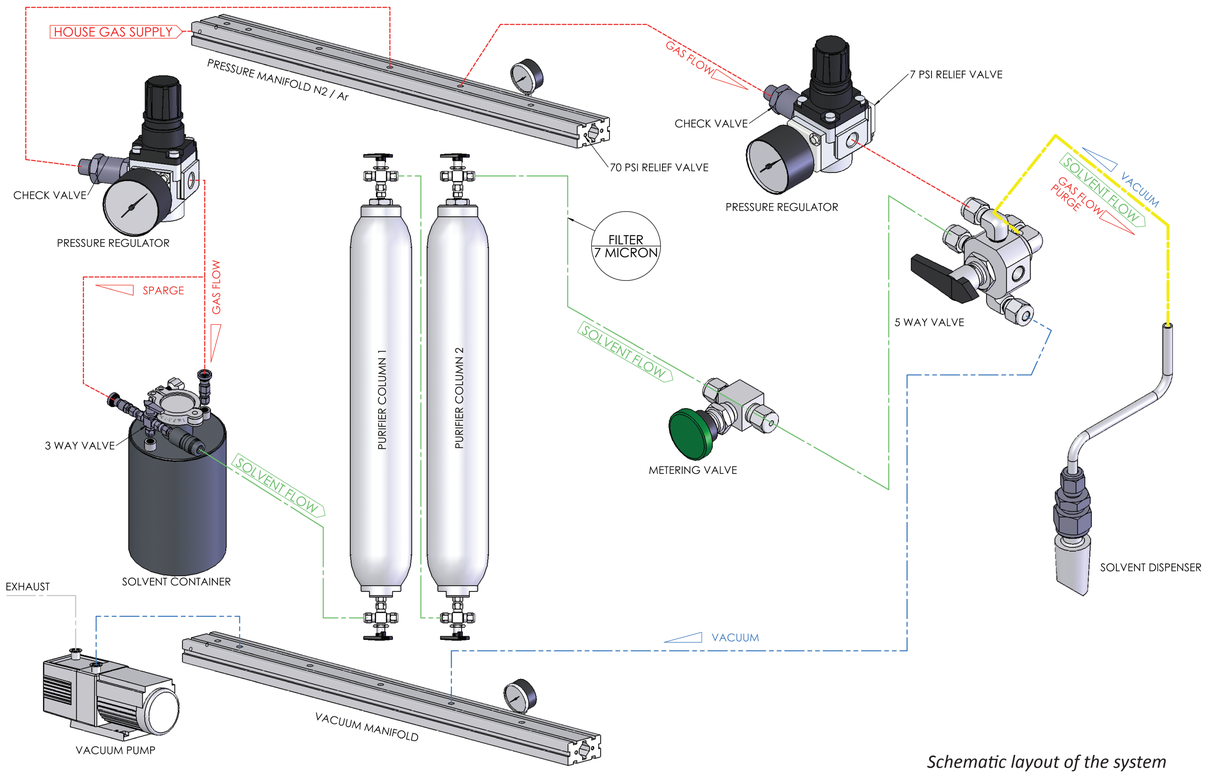
In order to obtain satisfactory results in many syntheses involving air moisture sensitive reactions, it may be necessary to purify solvents to remove reactive impurities such as water, other protic / acidic materials, or atmospheric contaminants such as oxygen. Traditionally, chemists and other laboratory workers who require extremely high purity solvents were forced to purify these solvents by reflux at elevated temperatures, over water reactive drying agents (Li, Na, K, CaH2, LiAlH4).

Over the years, there have been many fires and explosions involving thermal distillation apparatus. The consequence of these accidents are fires in the lab that seriously burn students, and caused millions in property damage.

In 1996, Pangborn published in the journal Organometallics an alternative method to thermal distillation. In 2001, Alaimo suggested modifications to the apparatus described by Pangborn in the Journal of Chemical Education. The so called "Grubbs apparatus" uses a large solvent reservoir and alumina/catalyst columns to dry and de-oxygenate solvents to the desired level of purity; without using heat or water-reactive drying agents.

HOW DOES THE "GRUBBS SYSTEM WORK?"

The Grubbs apparatus consists of; a stainless steel solvent reservoir (typically 18 litres), one alumina column, one catalyst on alumina column, and a purified solvent delivery port. The reservoir is pressurized (< 0,7 Bar) with an inert gas like Nitrogen or Argon and the solvent is pushed out of the reservoir through the columns to an evacuated glass flask or delivered directly to the experiment. Columns and reservoirs can be set up in parallel with carefully labelled valves to purify several solvents on demand. A vacuum pump maintains negative pressure on the manifold and collection flasks and exhausts inert gas at the end of the apparatus.



Schematic layout of the system

ADVANTAGES OF THE GRUBBS METHOD
(over traditional thermal distillation method).

If you want to work quickly, easy and above all SAFELY the Inert Solvent Purification Systems are your ideal solution. These systems are safe alternatives to thermal distillation, alleviating risk of injury to your staff or fire damage to your lab. They allow for air-free collection of ultra-dry and de-oxygenated solvents, while eliminating cross-diffusion of solvent vapours.

- New and improved ether, alcohol and amine drying columns and filtration design
- Simultaneous dispensing of up to 7 solvents on one system
- Safe alternative to thermal distillation method
- Free up valuable hood space
- Integration with a Glovebox to have direct access to solvents inside the box
- Low annual operating costs compared to thermal distillation method.
- The GRUBBS system does involve large quantities of solvent compaired to thermal distillationmethod.
- Purified solvent "from-the-tap". Several litres of ultra dry and de-oxygenated solvents are waiting to be collected from the GRUBBS system.
- The solvents are contained in metal cans, which are safer than glass bottles.

EXPECTED MOISTURE RESULTS USING HPLC SOLVENT

SOLVENT TYPE:	EXPECTED TITRATION RESULTS	ALUMINA	SIEVES	DE-GAS	COPPER
Acetonitrile	15 ppm	•		•	
Benzene	4 ppm	•		•	•
Chloroform	2 ppm	•		•	
Dichloromethane	2 ppm	•		•	
DME	30 ppm	•		•	
DMF	30 ppm		•	•	
Ether - Inhibitor-free	8 ppm	•		•	
Heptane	2 ppm	•		•	•
Hexane	2 ppm	•		•	•
Hexanes	2 ppm	•		•	•
Methanol	40 ppm	•		•	
Pentane	2 ppm	•		•	•
Chlorobenzene	6 ppm	•		•	
THF - Inhibitor-free	7 ppm	•		•	
Toluene	2 ppm	•		•	•

- • = Solvent will be dried using identified material.
- • = All Solvents must be de-gassed for oxygen removal using inert gas. Degas times are located in Manual.
- • = Identified solvents may be passed through Copper Catalyst for optimum oxygen scrubbing.

This table shows the moisture levels from different solvents after being purified with the BRUBBS solvent purification system.

AND HOW DOES THE GRUBBS SYSTEM LOOK

CONCLUSIONS

Distillation and purification of flammable solvents, using the solvent still method, will continue to be an integral part of chemical experimentation, if only on a limited basis as alternative methods become available. Active metals increase the likelihood of an explosion and fire with flammable liquids. In the past the solvent still method of purifying organic solvents had an acceptable level of risk because there was no alternative way of obtaining a moisture- and oxygen- free product.

New Solvent Purification Systems using the GRUBBS technology are an cost effective alternative way of obtaining a moisture- and oxygen-free product.