Labware Notes
Delivering the Tools for Science
Vol. 3, No. 2

SIGMA-ALDRICH Labware

Aldrich Kugelrohr – "Bulb-to-Bulb" Vacuum Distillation Apparatus

The German word *kugelrohr* translates to "bulb tube." In chemical synthesis, *kugelrohr* refers to a distillation apparatus where the "bulbs" are operated in a horizontal position. Bulb-to-bulb vacuum distillation is a useful laboratory technique for the purification of liquid and low melting solid substances, sensitive high boiling liquids, and quickly distills the most difficult materials with minimal holdup due to the very short distillation path. Consider the *Kugelrohr* technique when separation by crystallization is unsuccessful and distillation is impractical because of product solidification in the distillation apparatus.

Applications:

- Distills heat sensitive compounds
- Distills liquids and low melting solids from polymers and tars
- Simple fractionations when boiling points are 20 to 30 °C apart
- Semi-micro solvent evaporator
- Removes color and particulates
- Sublimation
- Dries inorganic salts
- Distills ionic liquids

Benefits of the *Kugelrohr* Technique:

- Simple to set up and yields rapid results
- Reduces distillation time up to 90% compared to conventional short path apparatus
- Minimal hold up and loss of product

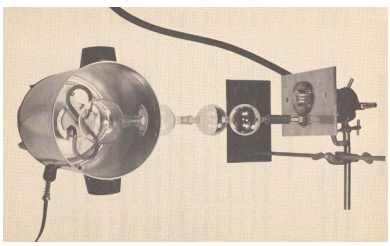

Figure 1. *In the early days of this technology, a modified coffee pot was used as a heater and a windshield wiper motor was connected to tube or pipe.*

Basic Principal of Operation:

The distilling flask (pot flask) is filled a third full, heated in an air bath, and the distillate collects in the next bulb located outside the air bath. A safe and simple cooling system that utilizes dry ice instead of flammable solvents to cool the bulbs can be used. Readily available plastic or cardboard cylindrical tubs of 4 to 5 cm in diameter and 10 cm in length are adapted to the shape of the bulb by making two circular cuts on opposite sides of the bottom of the cylinder. This increases the cooling surface on the bulb.

Figure 2. *The modern Aldrich Kugelrohr is adaptable, safe, and precise using flask sizes from 10 mL to 2 L and thermocouple temperature control up to 220 °C, +/-1 °C.*

Cleaning Tips for Top Performance:

During use, untrapped chemical vapors may pass through the rotating drive shaft of the vacuum drive unit, depositing solids in the small vacuum orifice that is located on the shaft just above the vacuum hose connection. A plugged orifice will reduce vacuum performance significantly. Usually this problem can be remedied by simply washing out the drive shaft with an appropriate solvent to remove the obstruction in the vacuum orifice. If the simple leaning procedure does not improve vacuum performance, then a thorough cleaning of the rotating shaft and vacuum seals is needed.

These cleaning procedures are detailed in Technical Information Bulletin AL-219.

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Paula's Pointers

Kugelrohr distillations require high vacuum

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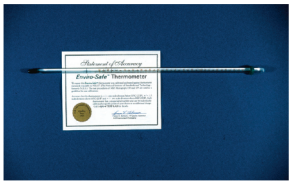
All vacuum distillations require the media that is being pumped to remain pure. This affects the choice of vacuum pump that is used. KNF Laboport solid PTFE vacuum pumps are a good choice for vacuum distillations. These are quiet, high-performance vacuum pumps capable of vacuum down to 1.5 torr. Manufactured from chemically inert components with an oil-free operation that ensures the pumped media stays pure.

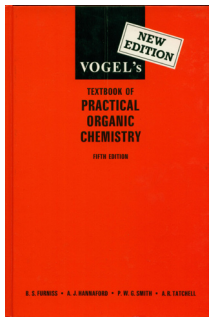
Z288209 115V AC, 60Hz
Z288217 230V AC, 50 Hz

Labware Listens



With the increasing emphasis to preserve the environment, we've had an increasing demand for "green" product options. One of the low environment impact ranges we have added is *Enviro-Safe*™ Thermometers. They are NIST calibrated, serialized and provided with Statement of Accuracy. Their non-hazardous, biodegradable, green liquid fill avoids all of the safety and disposal problems associated with mercury thermometers.

Background Reading



Z203947

Aldrich Vogel's Textbook of Practical Organic Chemistry, 5th Ed.

General description This edition retains the comprehensive, reference-text character, updated, with the introduction of new reagents and techniques as well as the development of a philosophy of organic synthesis, integrating mechanistic theory with the strategy and methodology of synthesis. Chapters deal with experimental techniques, and spectroscopic methods, as well as specific aliphatic, aromatic, alicyclic, heterocyclic, and organiccompounds.

Down Time

8	3		7	5				
			4			2	5	
1					6	3		
2	9	6						
	8							
							1	6
		7					8	1
6			9		7			
5	2					7		

Sudoku is a logic-based number placement puzzle. The objective is to fill a 9×9 grid so that each column, each row, and each of the nine 3×3 boxes contains the digits from 1 to 9 only one time each. Completed Sudoku puzzles are usually a type of Latin square with an additional constraint on the contents of individual regions. Sudoku means "single number" in Japanese.

Labware Links

For more detailed information on the products featured in this newsletter along with back issues and many useful Labware web links and protocols visit ***sigma-aldrich.com/labwarenotes***

Latest News

Aldrich has developed a new range of glassware kits containing complete distillation setups with threaded joints. These require no clamps and are non-contaminating because they are greaseless. The self-extracting design prevents the joints seizing during use.

- Threaded joints remove need for unreliable clamps
- Greaseless joints remove cause of contamination
- Self-extracting design prevents threads seizing

More details can be found at ***sigma-aldrich.com/labwarenotes***

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