SIGMA-ALDRICH
Labware NOTES
Delivering the Tools for Science


# The "Golden Rule" for Solvent Removal

Solvent removal remains an unavoidable process that scientists and engineers must perform on scales ranging from a few milliliters to thousands of liters. The use of a rotary evaporator in this process has become ubiquitous for single batch and continuous processes. Most scientists become familiar with the technique in a university experimental laboratory course. The technique can become trivial quickly – much like other daily tasks. Unfortunately many rotary evaporator users forget the scientific principles of solvent evaporation. This is evidenced by pulling ultimate vacuums without control or knowledge of the pressure, the use of secondary condensers, problematic bumping and the use of bump traps. To achieve optimal distillation conditions, the distillation energy supplied by the heating bath must be removed by the condenser. An easy concept to remember for solvent removal via rotary evaporation is the **20/40/60 Rule**. These numbers refer to the D20°C principle.

## 20/40/60 Rule

The vacuum is adjusted properly for each solvent with an operating bath temperature of 60°C to yield a solvent vapor temperature of 40°C which is subsequently condensed at 20°C. The rule can be applied to lower bath temperatures too, e.g.: Cooling water: 0°C; Vapor: 20°C; Bath: 40°C. Obviously an accurate and adjustable vacuum control system must be employed. The 20/40/60 Rule makes solvent removal simple and effective. The vacuum is the only setting to alter and that value is chosen easily from a chart.

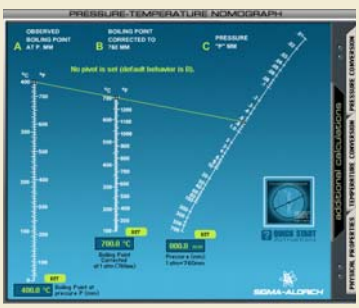
Since Buchi invented the Rotary Evaporator 50 years ago, Buchi has continued to make advancements to the Rotavap as well as vacuum pumps and controllers. The newest controllers from Buchi feature not only a large LCD display and easy-to-use interface, but some novel features as well. Buchi vacuum controllers feature a full solvent library showing all of the parameters for the reaction. The correct pressure can be automatically adopted from the solvent library. The new gradient function allows users to evaporate under a controlled pressure gradient. Another new feature


gives the ability to repeat manual distillations or save them as a program to be used later. The most advanced controller, the V855, even has the ability to perform an automatic distillation- one push of a button allows the user to perform a gentle, reproducible distillation that maximizes solvent recovery. Join these controllers with Buchi's newest line of pumps and you have an industry leading combination.

## 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.***

## Pressure-Temperature Nomograph




Our on-line Pressure-Temperature Nomograph is the ideal tool to help you quickly and easily estimate boiling points at various pressures. The interactive Pressure-Temperature Nomograph simplifies calculations to improve the efficiency of your distillation or evaporation. The Pressure-Temperature Nomograph is a graphical application of the Clausius-Clapeyron Equation, which assumes the heat of vaporization is a constant over a pressure range. Antoine's Equation gets around this by using empirical data for each unique liquid under consideration.

- Convert between five units of pressure using the built-in pressure conversion calculator
- Calculate temperature conversions
- Check physical data (650k PDF) for all your common solvents, including a list of relevant products.
- Calculate a boiling point or pressure using the Antoine Equation

# Paula's Pointers

**Protection from bumping samples during ro tary evaporation**

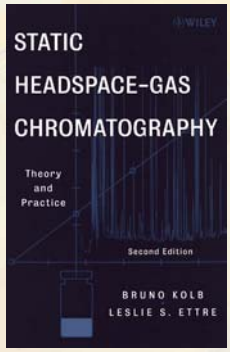
Technically a splash-guard or bump trap on your rotary evaporator is unnecessary. However protection from bumping is needed with problematic solvents such as ethyl acetate or where solids precipitate from the sample. Also in cases where the sample is precious or irreplacable these adapters can bring peace of mind for very little outlay. There are various styles, each having their own characteristics. Perhaps the most effective is the pear-shaped design. These eliminate the hold-up that is common to the round style of splash-guard adapters. The tapered sidewalls permit continuous washing with condensed solvent while preventing vapour cooling points along the path of the condenser. The increased volume of the trap prevents splashing or bumping and keeps solvent out of the vapour tube.

Visit ***sigma-aldrich.com/labwarenotes*** for full product details

# Background Reading

This second edition has been thoroughly updated to reflect the most recent developments and practices, and includesa chapter on methods for the complete evaporation of an analyte.

**Z705535** Static Headspace – Gas Chromatography: Theory and Practice




# Latest News

**Universal Rotary Evaporator Adapter for Jars and Bottles Coming Soon!**

An adapter for connecting wide-mouth glass jars and bottles to your rotary evaporator will be available soon from Sigma-Aldrich Labware. The adapter is made of glass-filled PTFE and features a unique "universal-fit" threaded connection for jar caps. The adapter includes one 58mm diameter holed cap and a solid cap with PTFE liners and borosilicate glass jars will be also available in convenient packs.

**Simple Installation**

Insert the threaded PTFE shaft of the adapter through the top of the jar cap and tghten the nut by hand. The other end of the adapter has a 24/40 or 29/32 female joint for connection to the evaporator vapor tube.

**Field Tested**

The adapter has been extensively tested for evaporating solvents from asphalt and petroleum fractions using a standard size jar instead of a round-bottom flask from which sample recovery can be difficult. The adapter will be equally convenient for the recovery of solid materials in other laboratory applications.

If you would like more information on availability please email us at ***labware@sial.com.***

# Labware Listens

Technical Service receives thousands of calls daily, some from customers trying to identify and order replacement parts for their trusty Buchi rotary evaporators or to replace a complete evaporator system. Often a particular Buchi configuration is required, i.e. evaporator, temperature controller, vacuum pump, and chiller. To enable customers to more easily locate these products we created the new Buchi Browser on our website. This browser brings all Buchi products together in a way that simplifies product identification and selection and even includes a listing of parts by Buchi number. To view the Buchi Browser go to: ***sigma-aldrich.com/labwarenotes.***

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

# Down Time

Number puzzles first appeared in newspapers in the late 19th century in the form of magic squares which relied on arithmetic to solve. Soon after a Suduko like puzzle appeared. Unlike the Magic Square, Suduko is a logic based puzzle where the objective is to complete the grid so that each column, row and each 3x3 square contains the digits 1 to 9. The completed puzzles are a form of Latin square with the addition of no repetitions in the 3x3 square.



JVX
03087-504402
0087