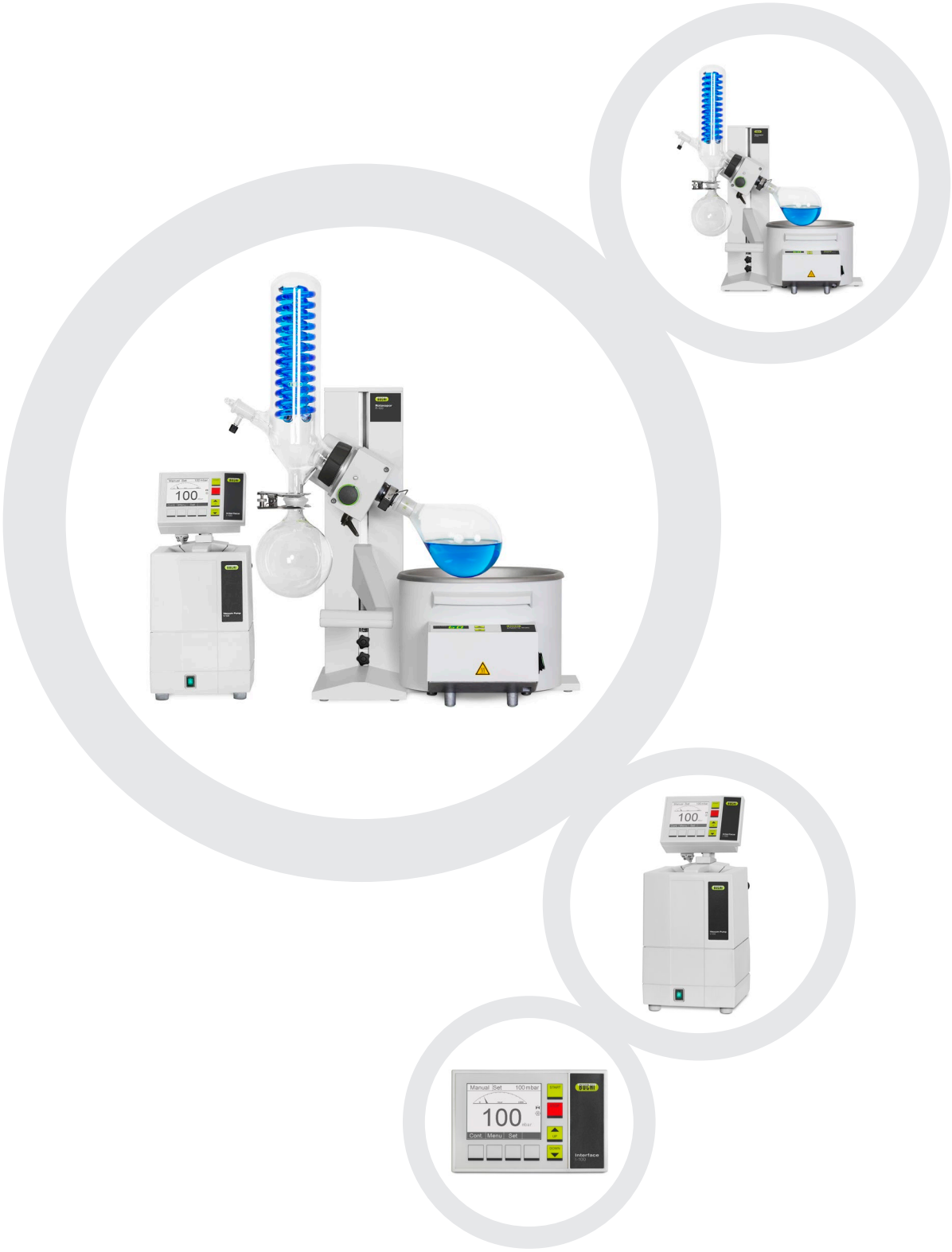
Application Note
No. 198/2015

Quality improvement of common solvents by distillation using the Rotavapor® R-100

Distillation System Rotavapor® R-100, I-100, V-100



1. Introduction

In chemical laboratories, especially in synthetic, analytical and pharmaceutical laboratories, a wide variety of solvents are applied. Obviously, the solvent quality, its environmental and health impact have to be carefully considered before use.
The cost of the solvents is directly proportional to its purity, i.e. the higher the purity the higher the costs. However, even in highly pure solvents, residual impurities can either impact the synthesis of a compound itself. Furthermore the impurities can be a great challenge for interpreting analytic results as for example NMR-data.
In a simple study, combining rotary evaporation and gaschromatography with flame ionisation detection, GC-FID analysis, we present the impact of single and double distillation of high purity Chinese solvents. Six common organic solvents have been single and double distilled using an R-100 System.
It is revealed that distillation prior to use may lead to even purer solvents and probably, higher syntethic yields and simpler post analysis.

2. Equipment

- Rotavapor® R-100 with the BUCHI Vacuum Pump V-100
- GC-FID (Agilent 7890)

3. Chemicals

Chemicals:

- Ethanol, Purity > 99.7 %, Tianjin guangfu fine chemical research institute
- Acetone, Purity ≥ 99.5 %, Beijing chemical works
- Hexane, Purity ≥ 99.5 %, Tianjin guangfu fine chemical research institute
- Toluene, Purity ≥ 99.5 %, Beijing chemical works
- Acetonitrile, Purity ≥ 99.5 %, Tianjin fuchen chemical reagents factory
- Dichloromethane, Purity > 99.5 %, Tianjin guangfu fine chemical research institute

For a safe handling please pay attention to all corresponding MSDS.

4. Procedure

4.1 Solvent distillation

The following steps were applied to distill the solvents:

1. Make sure that the heating bath is filled with distilled water
2. Ensure that the glassware used is scrupulously clean
3. Switch on the heating bath and vacuum pump
4. Open the tap water, make sure the flow rate is at least 40 L/h
5. Pour 100 mL solvent into evaporating flask
6. Make sure that the receiving flask is fitted
7. Set the vacuum and bath parameters listed in Table 1

Table 1: Parameters for the Rotavapor® R-100

Solvent	Ethanol	Hexane	Acetonitrile	Acetone	Dichloromethane	Toluene
Pressure setting	80 mbar	260 mbar	125 mbar	260 mbar	550 mbar	43 mbar
Water bath temperature	45 °C	45 °C	45 °C	45 °C	30 °C	45 °C

8. Switch on the rotary drive unit and adjust the rotation speed to level 3-4

9. Immerse the evaporating flask in the heating bath
10. Wait 1 – 2 minutes to make sure distillation starts
11. When a few distilled solvent dropped into the receiving flask, vent the vacuum and rinse the receiving flask with the distilled solvent
12. Start the vacuum unit again go on distilling the rest of solvent
13. Take 1 mL of the distilled solvent to the sample vial for further analysis via Gas Chromatography when the first distillation is finished
14. For the second distillation pour the first distilled solvent to the evaporating flask and start the distillation again following the steps 7 - 12

4.2 GC-FID conditions

- Column: HP-5, 325 °C, 30 m × 250 µm × 0.25 µm
- Flow rate: 1 mL/min
- Injection volumn: 1 µL
- Carrier gas: N2
- Oven: starting at 50 °C and holding for 1 min; increasing at 5 °C/min to 100 °C; increasing at 20 °C/min to 250 °C and holding for 1 min. the total running time is 19.5 min
- Detector: FID

5. Results and Discussion

The solvents were analyzed as received as well as after first and second distillations by GC-FID. Chromatographic comparison among the solvents are shown in Figure 1. Importantly, we show only sections of the chromatogram that do not interfere with the solvent signal itself and that we tentatively assign to solvent impurities, without further characterisation.

Probably, the impurities are volatile compounds contained in the solvents in very low quantities. Such impurities could originate from the production process of the solvent. For these impurity signals, the signal areas bewteen the crude solvent and the distilled solvent are summarized in table 1 - 6.

From figure 1, we immediately see that the distillation can greatly improve the purity of the solvent and decrease the base line noise. For hexane, acetone, and acetonitrile a single distillation can effectively reduce the area of impurity peaks.

For all solvents, the second distillation reduces the area of impurity more than 50 %, compared to before distillation. Clearly, the exact quantification of the impurities is only possible when using a proper internal standard and a decent linear calibration. However, here we show that the purity of the distilled solvents greatly increase by distillation with the by Rotavapor® R-100.

Hence, this simple study confirms that the distillation of already pure solvent using a rotary evaporator is beneficial to further increase solvent purity.

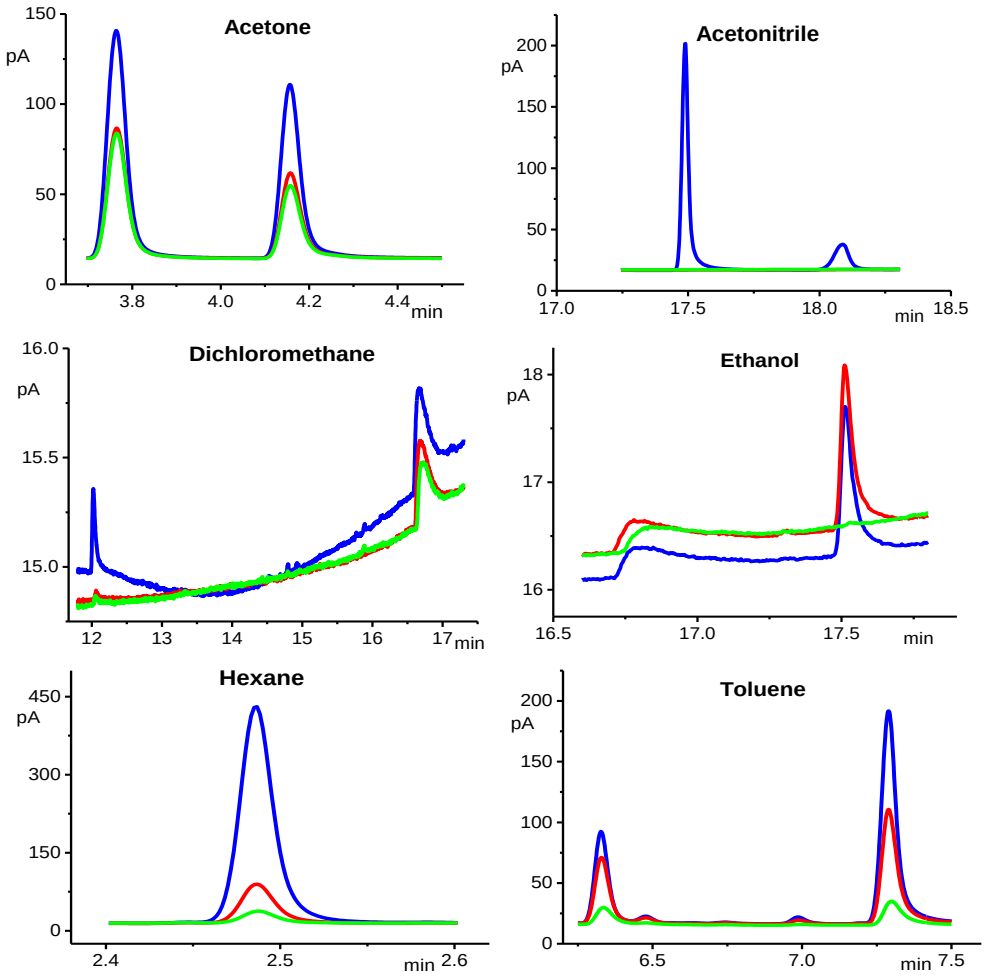


Figure 1: the comparison of six solvents - the blue line refers to the solvent as received; the red line refers to the 1st distilled solvent and the green line refers to the 2nd ditillded slovent). For acetonitrile the red lind and the green line are identical, thus only the green line is shown.

*Table 2: comparison of impurity peak area in **acetone***

RT [min]	The crude [Area,]	1st distillation [Area]	2nd distillation [Area]
3.76	407.3	243.4	235.3
4.16	316.7	163.5	141.5
Percentage of the first peak area [%]	100	59.8	57.8
Percentage of the second peak area [%]	100	51.6	44.7

*Table 3: comparison of impurity peak area in **acetonitrile***

RT [min]	The crude [Area]	1st distillation [Area]	2nd distillation [Area]
17.49	347.9	ND	ND*
18.09	88.03	ND	ND*
Percentage of the first peak area [%]	100	0	0
Percentage of the second peak area [%]	100	0	0

*ND means NOT Detected

*Table 4: comparison of impurity peak area in **dichloromethane***

RT [min]	The crude [Area]	1st distillation [Area]	2nd distillation [Area]
12.00	1.322	0.146	0.114
16.67	5.591	4.682	2.963
Percentage of the first peak area [%]	100	11.0	8.6
Percentage of the second peak area [%]	100	83.7	53.0

*Table 5: comparison of impurity peak area in **ethanol***

RT [min]	The crude [Area]	1st distillation [Area]	2nd distillation [Area]
16.81	2.537	2.072	1.108
17.51	4.693	4.463	ND*
Percentage of the first peak area [%]	100	81.7	43.7
Percentage of the second peak area [%]	100	95.1	0

ND means NOT Detected

*Table 6: comparison of impurity peak area in **hexane***

RT [min]	The crude [Area]	1st distillation [Area]	2nd distillation [Area]
2.49	577.2	109.6	30.6
Percentage of peak area [%]	100	19.0	5.3

*Table 7: comparison of impurity peak area in **toluene***

RT [min]	The crude [Area]	1st distillation [Area]	2nd distillation [Area]
6.33	265.6	194.3	62.9
7.29	699.1	404.0	99.9
Percentage of the first peak area [%]	100	73.2	23.7
Percentage of the second peak area [%]	100	57.8	14.3

6. Conclusion

It is shown that the Rotavapor® R-100 is successfully applied in improving the solvent purity and removing volatile organic components, even in already pure solvents.
Selected impurities were analysed by GC-FID in six common organic solvnets with a declared puritiy of more than 99.5 %. A 50 % reduction of the observed peak areas after maxium two distillation showed the benefits distilling the solvents with a Rotavapor®. Clearly, lower impurity levels reduce the risk for contaminations during synthesis, may enhance the yield and simplify the analytical work-up and analysis. Furtermore, an even greater purification effect is expected Solvent distillation using a Rotavapor® could significantly save cost when upgrading low purity solvent.

7. Acknowledgement

We greatfully acknowledge Professor Qiu Jing from the Institute of Quality Standards & Testing Technology for Agro-Products of CAAS, Beijing China, for his support in GC-FID analysis and sharing the results.