Supporting Information for:

Static Fluid Condensers for the Containment of Refluxing Solvent

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Table of Contents

Glassware Part Order Information

For 24/40 adapter: Part number - DOW-1110-121GC. This part is available to any buyer from Chemglass Life Sciences. Description - Condenser, Allihn, 24/40 Joint, 300mm Jacket Length, 13-425 GPI Thread Near the Top, 45° Angle, Glass Only, Referenced from CG-1206-HC-05. For 14/20 adapter: Part number - Dow-1110-122GC; Description - Condenser, Allihn, 14/20 Joint, 110mm Jacket Length, 13-425 GPI Thread Near the Top, 45° Angle, Glass Only, Referenced from CG-1206-HC-05. SURE-Link™ septa from Chemglass Life Sciences were utilized for this purpose. Part number – CG-4910-15.

Medium Volume, 100 mL, Gravimetric Experimental Procedure.

To a pre-weighed 200 mL round-bottomed flask equipped with a Teflon®-coated stir bar and a plastic cap was added the desired solvent (100 mL) *via* graduated cylinder. The cap was replaced and the flask was re-weighed. The cap was removed and the flask equipped with a 300 mm jacket length 24/40 modified Allihn static ethylene glycol condenser with no nitrogen atmosphere. The flask was placed into a 200 mL aluminum block which was previously equilibrated to the desired temperature. Upon visual observation of gentle reflux, the experiment was allowed to proceed for 1 hour. After 1 hour, the flask was removed from the block and allowed to cool to room temperature. Following removal of the condenser, the flask was weighed for a final time. The following tables (S1-S6) outline the data obtained from individual solvents.

Table S1. Diethyl Ether

Run	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent T_F (g)	T_0 Mass (g)	T_F Mass (g)	Solvent Loss (g)	% Solvent Loss
1	153.03	223.43	222.86	70.40	69.83	0.57	0.81
2	157.51	227.76	227.29	70.25	69.78	0.47	0.67
3	155.91	226.14	225.76	70.23	69.85	0.38	0.54

Table S2. Pentane

Run	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent T_F (g)	T_0 Mass (g)	T_F Mass (g)	Solvent Loss (g)	% Solvent Loss
1	153.02	214.03	213.64	61.01	60.62	0.39	0.64
2	157.50	218.66	218.25	61.16	60.75	0.41	0.67
3	155.91	216.58	216.25	60.67	60.34	0.33	0.54

Table S3. Dichloromethane

Run	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent T_F (g)	T_0 Mass (g)	T_F Mass (g)	Solvent Loss (g)	% Solvent Loss
1	283.67	283.34	130.64	130.64	130.31	0.33	0.25
2	288.49	288.24	130.96	130.96	130.71	0.25	0.19
3	287.01	286.75	131.09	131.09	130.83	0.26	0.20

Table S4. Tetrahydrofuran

Run	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent T_F (g)	T_0 Mass (g)	T_F Mass (g)	Solvent Loss (g)	% Solvent Loss
1	153.03	239.41	239.30	86.38	86.27	0.11	0.13
2	157.48	243.20	243.07	85.72	85.59	0.13	0.15
3	155.91	242.80	242.69	86.89	86.78	0.11	0.13

Table S5. Xylenes

Run	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent T_F (g)	T_0 Mass (g)	T_F Mass (g)	Solvent Loss (g)	% Solvent Loss
1	155.17	281.66	281.57	126.49	126.40	0.05	0.06
2	159.59	289.14	289.06	129.55	129.47	0.05	0.06
3	158.01	287.22	287.14	129.21	129.13	0.05	0.06

Table S6. 1,2-Dichlorobenzene

Run	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent T_F (g)	T_0 Mass (g)	T_F Mass (g)	Solvent Loss (g)	% Solvent Loss
1	155.17	281.66	281.57	126.49	126.40	0.09	0.07
2	159.59	289.14	289.06	129.55	129.47	0.08	0.06
3	158.01	287.22	287.14	129.21	129.13	0.08	0.06

Small Volume, 10 mL, Gravimetric Experimental Procedure.

To a pre-weighed 25 mL round-bottomed flask equipped with a Teflon®-coated stir bar and a plastic cap was added the desired solvent (10 mL) *via* syringe. The cap was replaced and the flask was re-weighed. The cap was removed and the flask equipped with a 110 mm jacket length 14/20 modified Allihn static ethylene glycol condenser with no nitrogen atmosphere. The flask was placed into a 25 mL aluminum block which was previously equilibrated to the desired temperature. Upon visual observation of reflux, the experiment was allowed to proceed for 1 hour. After 1 hour, the flask was removed from the block and allowed to cool to room temperature. Following the removal of the condenser, the flask was weighed for a final time. Table S7 outlines the data obtained from these experiments.

Table S7. Small Volume Study, 10 mL, After 1 hour at Reflux

Solvent	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent T_F (g)	T_0 Mass (g)	T_F Mass (g)	Loss (g)	% Loss
Diethyl ether	27.6529	34.4885	34.1260	6.8356	6.4731	0.3625	5.3
Pentane	29.1190	35.4780	35.1230	6.3590	6.0040	0.3550	5.6
Dichloromethane	27.6530	40.8730	40.5930	13.2200	12.9400	0.2800	2.1
Tetrahydrofuran	29.1120	37.9080	37.7850	8.7960	8.6730	0.1230	1.4
Xylenes	27.6590	36.0420	36.0000	8.3830	8.3410	0.0420	0.5
1,2-Dichlorobenzene	29.1120	42.0340	41.9590	12.9220	12.8470	0.0750	0.6

Comparative Study of Condensers, 10 mL, Gravimetric Experimental Procedure.

To 3 pre-weighed 25 mL round-bottomed flasks equipped with a Teflon®-coated stir bars and plastic caps was added the desired solvent (10 mL) *via* syringe. The caps were replaced and the flasks were re-weighed. The caps were removed and the flasks equipped with either a 110 mm jacket length 14/20 modified Allihn static ethylene glycol condenser, 125 mm 14/20 Vigreux condenser, or 105 mm jacket length 14/20 water jacketed Liebig condenser under an atmosphere of nitrogen. The flasks were placed into separate 25 mL aluminum blocks which

were previously equilibrated to the desired temperature. Upon visual observation of gentle reflux, the experiments were allowed to proceed for 18 hours. After 18 hours, the flasks were removed from the bath and allowed to cool to room temperature. Following removal of the condensers, the caps were replaced and the flasks were weighed for a final time. The following tables (S8-S10) outline the data obtained from this study.

Table S8. Comparative Study of Condensers, 110 mm Allihn Static Condenser

Solvent	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent[a] T_F (g)	T_0 Mass (g)	T_F Mass (g)	Loss (g)	% Loss
Diethyl ether	34.5704	41.5484	34.5704	6.9780	0.0000	6.9780	100.0
Dichloromethane	34.5704	47.9486	41.4434	13.3782	6.8730	6.5052	48.6
Tetrahydrofuran	34.5923	43.4220	39.8064	8.8297	5.2141	3.6156	40.9
Acetonitrile	34.5913	42.2988	41.8414	7.7075	7.2501	0.4574	5.9
Acetonitrile[b]	34.5798	42.2682	39.2160	7.6884	4.6362	3.0522	39.7
Xylenes	34.5932	43.1076	42.2282	8.5144	7.6350	0.8794	10.3
Xylenes[b]	34.5737	43.1156	39.8179	8.5419	5.2442	3.2977	38.6
1,2-Dichlorobenzene	34.5826	47.3615	46.9017	12.7789	12.3191	0.4598	3.6

[a]Flasks that appeared empty were not weighed and the flask mass was assumed. [b]Vigorous refluxing.

Table S9. Comparative Study of Condensers, 125 mm Vigreux Condenser

Solvent	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent[a] T_F (g)	T_0 Mass (g)	T_F Mass (g)	Loss (g)	% Loss
Diethyl ether	34.5664	41.5540	34.5664	6.9876	0.0000	6.9876	100.0
Dichloromethane	34.5664	47.6672	34.5664	13.1008	0.0000	13.1008	100.0
Tetrahydrofuran	34.5838	43.3806	34.5838	8.7968	0.0000	8.7968	100.0
Acetonitrile	32.4944	40.1757	37.9399	7.6813	5.4455	2.2358	29.1
Acetonitrile[b]	34.5754	42.3014	34.5754	7.7260	0.0000	7.7260	100.0
Xylenes	32.4936	41.0576	39.4936	8.5640	7.0000	1.5640	18.3
Xylenes[b]	34.5623	43.0596	40.7170	8.4973	6.1547	2.3426	27.6
1,2-Dichlorobenzene	34.5671	47.3290	46.7350	12.7619	12.1679	0.5940	4.7

[a]Flasks that appeared empty were not weighed and the flask mass was assumed. [b]Vigorous refluxing.

Table S10. Comparative Study of Condensers, 105 mm Water Jacketed Liebig

Solvent	Flask (g)	Flask + Solvent T_0 (g)	Flask + Solvent[a] T_F (g)	T_0 Mass (g)	T_F Mass (g)	Loss (g)	% Loss
Diethyl ether	32.4760	39.4051	32.4760	6.9291	0.0000	6.9291	100.0
Dichloromethane	32.4760	45.4281	39.9545	12.9521	7.4785	5.4736	42.3
Tetrahydrofuran	32.5042	41.3266	38.0204	8.8224	5.5162	3.3062	37.5
Acetonitrile	34.5848	42.3128	41.8224	7.7280	7.2376	0.4904	6.3
Acetonitrile[b]	32.4853	40.1732	38.5678	7.6879	6.0825	1.6054	20.9
Xylenes	34.5857	43.0739	42.0517	8.4882	7.4660	1.0222	12.0
Xylenes[b]	32.4813	41.0447	39.5917	8.5634	7.1104	1.4530	17.0
1,2-Dichlorobenzene	32.4840	45.2978	44.7351	12.8138	12.2511	0.5627	4.4

[a]Flasks that appeared empty were not weighed and the flask mass was assumed. [b]Vigorous refluxing.

Large Volume, 200-250 mL, Volumetric Experimental Procedure.

To a pre-weighed 500 mL round-bottomed flask equipped with a Teflon®-coated stir bar was added the measured desired solvent (250 mL) *via* a graduated cylinder. The flask was equipped with a 300 mm jacket length 24/40 modified Allihn static ethylene glycol condenser and placed under an atmosphere of nitrogen. The flask was heated on a 500 mL aluminum block which was previously equilibrated to the desired temperature. Upon visual observation of reflux, the experiment was allowed to proceed for 18 hours. After 18 hours, the flask was removed from the bath and allowed to cool to room temperature. Following removal of the condenser, the contents of the flask were poured into a graduated cylinder for a final visual measurement. Tables S11 outlines the data obtained from this study.

Table S11. Large Volume Study, 250 mL, After 18 hours at Reflux

Solvent	1	2	**3**	1	2	**3**	Average	%
	Final	Final	Final	Loss	Loss	Loss	(mL)	Loss

	Vol (mL)	Vol (mL)	Vol (mL)	(mL)	(mL)	(mL)		
Pentane	235	235	232	15	15	18	234	6.4
Diethyl ether	239	240	235	11	10	15	238	4.8
Dichloromethane	245	242	245	5	8	5	244	2.4
Tetrahydrofuran	245	230	250	5	20	0	242	3.3
Acetonitrile	237	235	250	13	15	0	241	3.7
Xylenes	250	250	249	0	0	1	250	0.1
1,2-Dichlorobenzene[a]	200	200	200	0	0	0	200	0.0

[a]Starting volume was 200 mL.