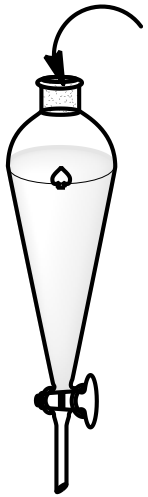
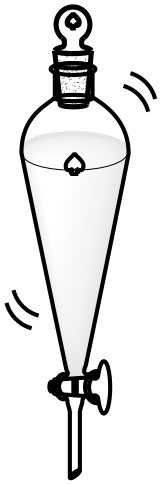
Extraction method

Protocol (liquid-liquid)

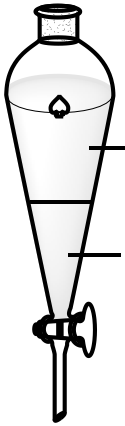
About 3 times



Separatory funnel



Mixing and release of pressure



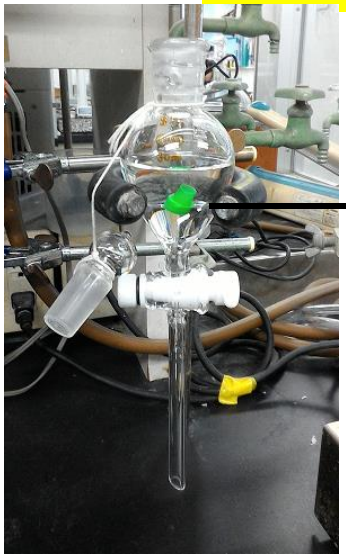
Separated

Halogenated solvent will be the lower layer.

Organic layer

Aqueous layer

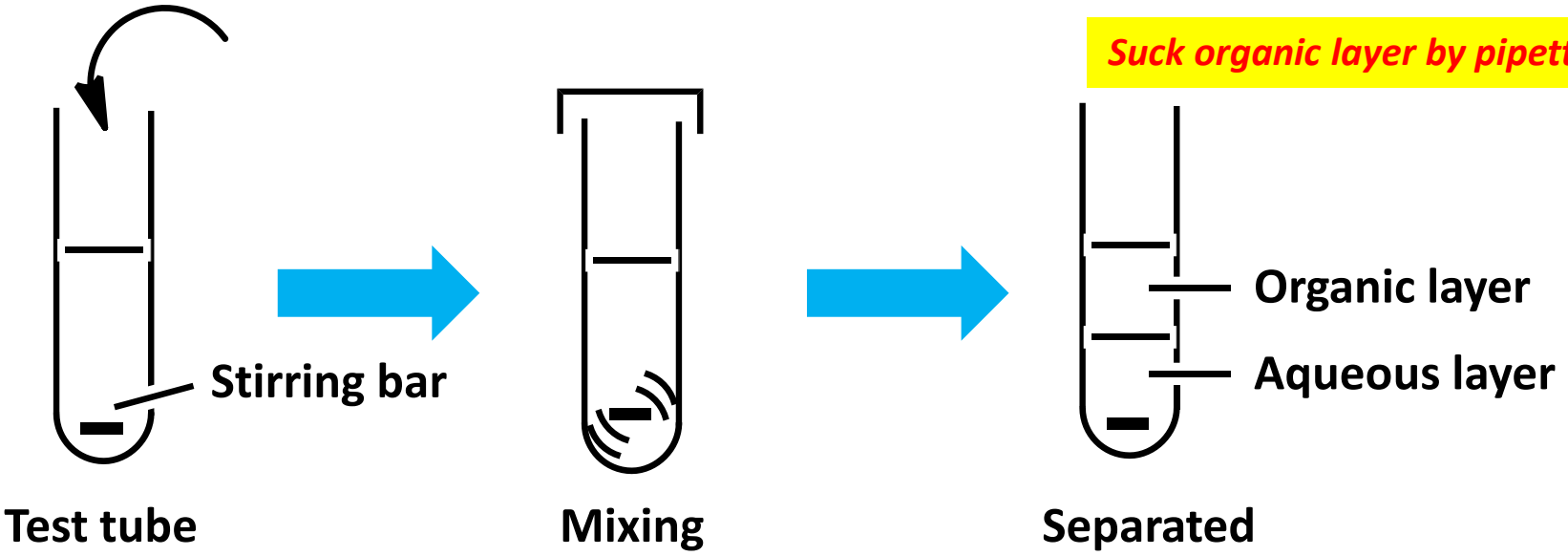
The obscure interface is checked by adding water or NMR tube and lighting.



NMR tube

Extraction method

Protocol (liquid-liquid, small scale)



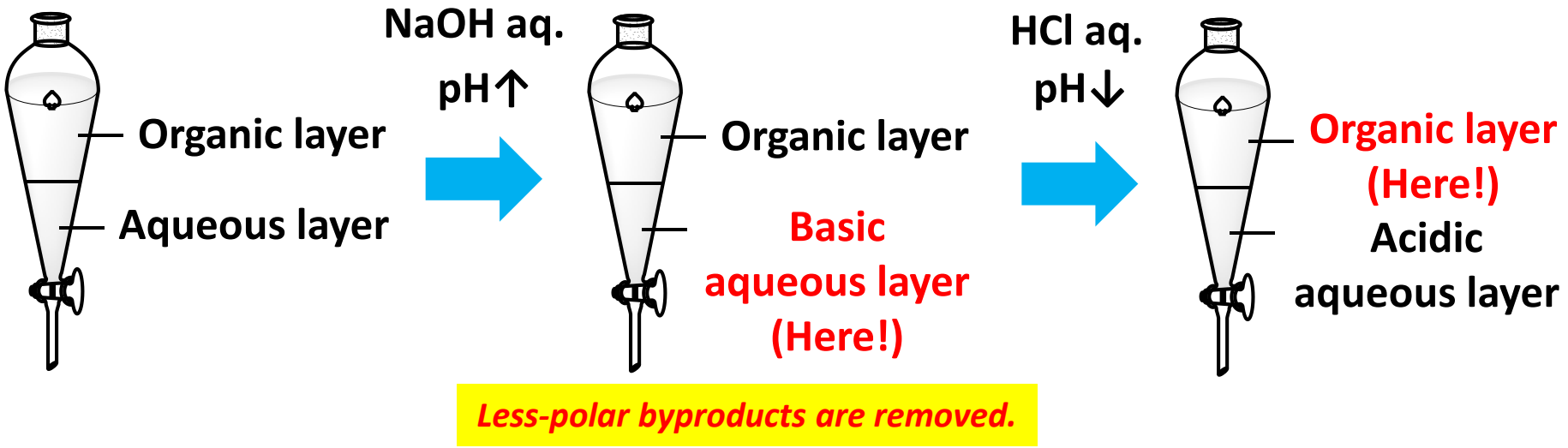
Tips

1. To check completion of the extraction, analyze the aqueous layer by TLC.
2. To extract highly polar products, use CH_2Cl_2:MeOH (9:1) or THF after adding brine to the aqueous layer or evaporate the aqueous layer.
3. To avoid emulsions, gently shake and swirl the funnel.
4. To destroy emulsions, add brine or hexane to the mixture or filter out impurities by Celite.
5. To remove DMSO or DMF, use hexane/EtOAc (3/1) or Et_2O.

Extraction method

6. To remove less-polar byproducts, firstly dissolve a product to aqueous layer and then to organic layer again by adjusting the pH of aqueous layer.

Ex.) Back extraction of carboxylic acid



Less-polar byproducts are removed.

Data of common solvents

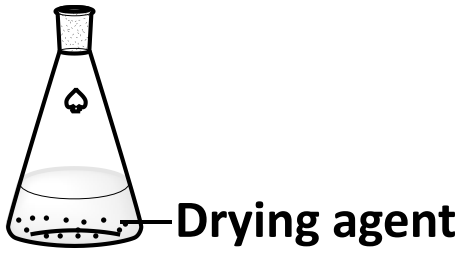
The organic solvent should

1. **Dissolve a product to be extracted.**
2. **Not react with a product to be extracted.**
3. **Not react with or be miscible with water.**
4. **Have a low boiling point.**

solvent	CAS	PRTR	M.W.	bp/℃	mp/℃	d	Et	affinity of water	class
1-butanol	[71-36-3]		74.12	117.7	-88.6	0.810	50.2	00	Protic
1-propanol	[71-23-8]		60.10	97.2	-126.2	0.804	50.7	000	Protic
1,2-dichloroethane	[107-06-2]	*116*	98.96	83.5	-35.7	1.252	41.9	0	Aprotic Polar
1,4-dioxane	[123-91-1]	*113*	88.11	101.3	11.8	1.034	36.0	000	Aprotic Polar
2-butanol	[78-92-2]		74.12	99.5	-114.7	0.807	47.1	00	Protic
2-butanone	[78-93-3]		72.11	79.6	-86.7	0.805	41.3	00	Aprotic Polar
2-methoxyethanol	[109-86-4]	*45*	76.10	124.6	-85.1	0.965	52.3	000	Protic
2-propanol (IPA)	[67-63-0]		60.10	82.2	-88.0	0.785	48.6	000	Protic
acetic acid	[64-19-7]		60.05	117.9	16.7	1.050	51.2	000	Protic
acetic anhydride	[108-24-7]		102.09	140.0	-73.1	1.083	3.9	0	Aprotic Polar
acetone	[67-64-1]		58.08	56.1	-97.4	0.790	42.2	000	Aprotic Polar
acetonitrile	[75-05-8]	*12*	41.05	81.6	-43.8	0.782	46.0	000	Aprotic Polar
benzene	[71-43-2]	*294*	78.12	80.1	5.5	0.879	34.5	0	Aprotic NonPol
carbon disulfide	[75-15-0]	*239*	76.13	46.2	-111.6	1.263	32.6	0	Aprotic Nonpol
carbon tetrachloride	[56-23-5]	*112*	153.84	76.6	-22.8	1.594	32.5	0	Aprotic Nonpol
chloroform	[67-66-3]	*95*	119.37	61.2	-63.5	1.489	39.1	0	Aprotic Polar
cyclohexane	[110-82-7]		84.16	80.7	6.7	0.779	32.1	0	Aprotic Nonpol
dichloromethane	[75-09-2]	*145*	84.93	39.6	-94.9	1.326	41.1	0	Aprotic Polar
diethyl ether	[60-29-7]		74.12	34.4	-116	0.714	34.6	0	Aprotic Polar
diisopropyl ether	[108-20-3]		102.18	68.5	-85.5	0.724	34.0	0	Aprotic Polar
dimethyl acetoamide	[127-19-5]		87.12	164.5	-20	0.937			Aprotic Polar
DME	[110-91-4]		90.12	84.5	-69	0.869	38.2	000	Aprotic Polar
DMF	[68-12-2]	*171*	73.09	153	-60.4	0.949	43.8	00	Aprotic Polar
DMSO	[67-88-5]		78.13	189.0	18.5	1.100	45.0	00	Aprotic Polar
ethanol	[64-17-5]		46.07	78.3	-114.5	0.789	51.9	000	Protic
ethyl acetate	[141-78-6]		88.10	77.1	-83.6	0.901	38.1	00	Aprotic Polar
ethylene glycol	[107-21-1]	*43*	62.07	197.5	-12.6	1.114	56.6	000	Protic
hexane	[110-54-3]		86.18	68.7	-95.3	0.659	30.9	0	Aprotic Nonpol
HMPA	[680-31-9]		179.20	233	7.2	1.027	40.9	000	Aprotic Polar
isobutylalcohol	[75-65-0]		74.12	107.9	-108	0.802		00	Protic
methanol	[67-56-1]		32.04	64.5	-97.7	0.791	55.5	000	Protic
nitromethane	[75-52-5]		61.04	101.2	-28.6	1.138	46.3	0	Aprotic Polar
o-xylene	[95-47-6]		106.17	144.4	-25.2	0.880		0	Aprotic Nonpol
pentane	[109-66-0]		72.15	36.1	-129.7	0.626		0	Aprotic Nonpol
propionitrile	[107-12-0]		55.08	97	-93	1.3660	43.7		Aprotic Polar
pyridine	[110-86-1]	*257*	79,10	115.3	-41.6	0.983	40.2	000	Aprotic Polar
tert-butanol	[78-83-1]		74.12	82.3	25.6	0.781	43.9	000	Protic
THF	[109-99-9]		72.11	66.0	-108.4	0.889	37.4	000	Aprotic Polar
toluene	[108-88-3]	*225*	92.14	110.6	-95.0	0.867	33.9	0	Aprotic Nonpol

Dehydration method

Protocol



Then filtered

or

Aspirator can be used.

Ex.) Data of drying agent in descending order of drying capacity

Na_2SO_4 (1.2 g H_2O/g dessicant) > $MgSO_4$ (0.2-0.8 g H_2O/g dessicant)
≧ $CaCl_2$ (0.2 g H_2O/g dessicant)

http://www.nacalai.co.jp/information/trivia2/01.html