



# General Chemistry Laboratory

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## Qualitative Analysis of Group 1 Cations



# Preparation

- Put on your lab coat and safety goggles
- Turn off your mobile phone
- Place your backpack in the drawer or the cabinet
- Put your prelab on lab bench (hold it down with something heavy) for ATA to sign

## Collect the following items

- Five centrifuge tubes, test tube tong
- Dropping pipettes
- Sticky labels
- NBR gloves
- Centrifuge (under lab bench)

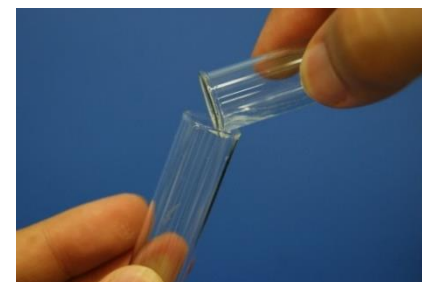


# Objective and Principles

- **Objective:** Separate and identify common Group 1 cations from a mixture solution based on the concepts of precipitation, dissolution, and formation of complex ions
- **Lab techniques:**
  - Systematic analysis of cations
  - Vortex mixer
  - Operating a centrifuge
  - Decantation
  - Using litmus paper to determine pH



**Vortex mixer**

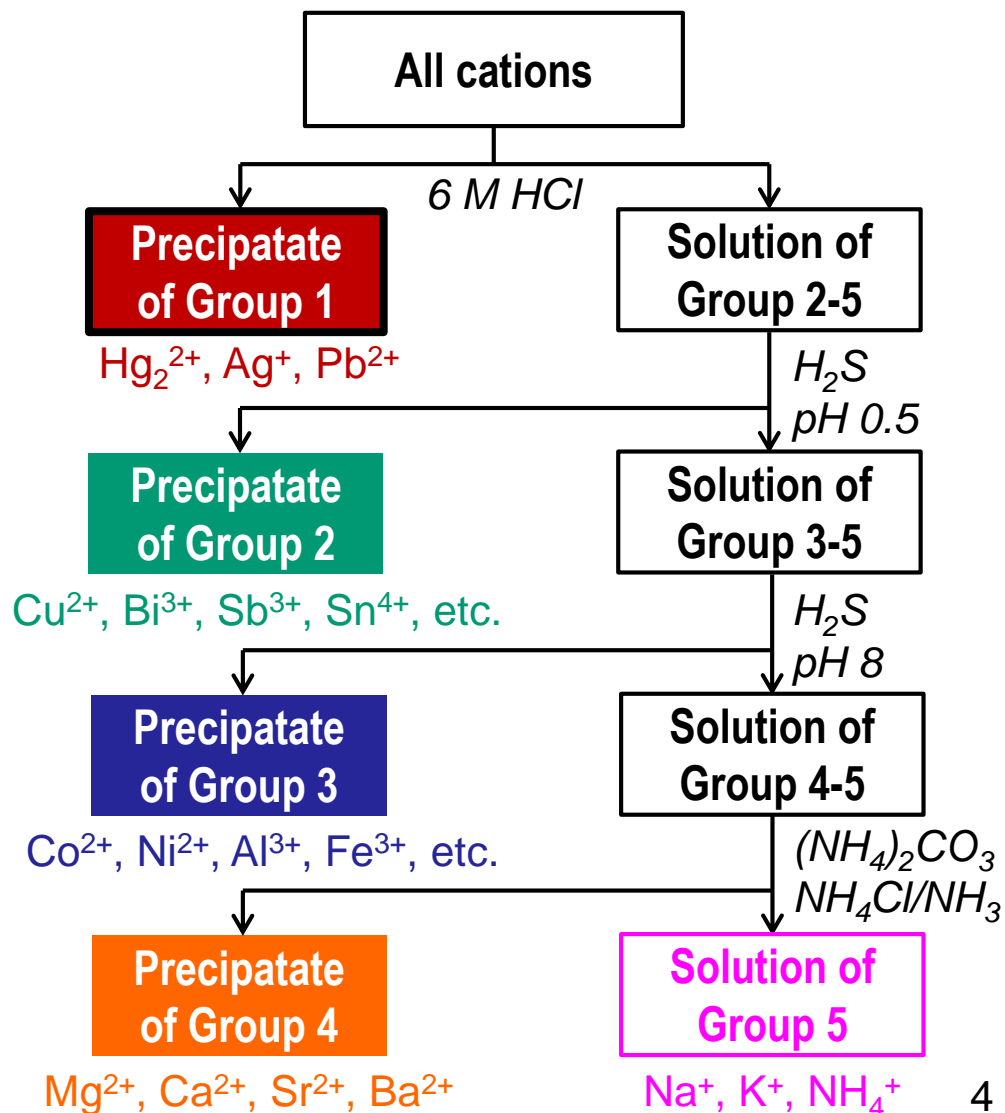


**Decantation**



# Qualitative Analysis of Cations

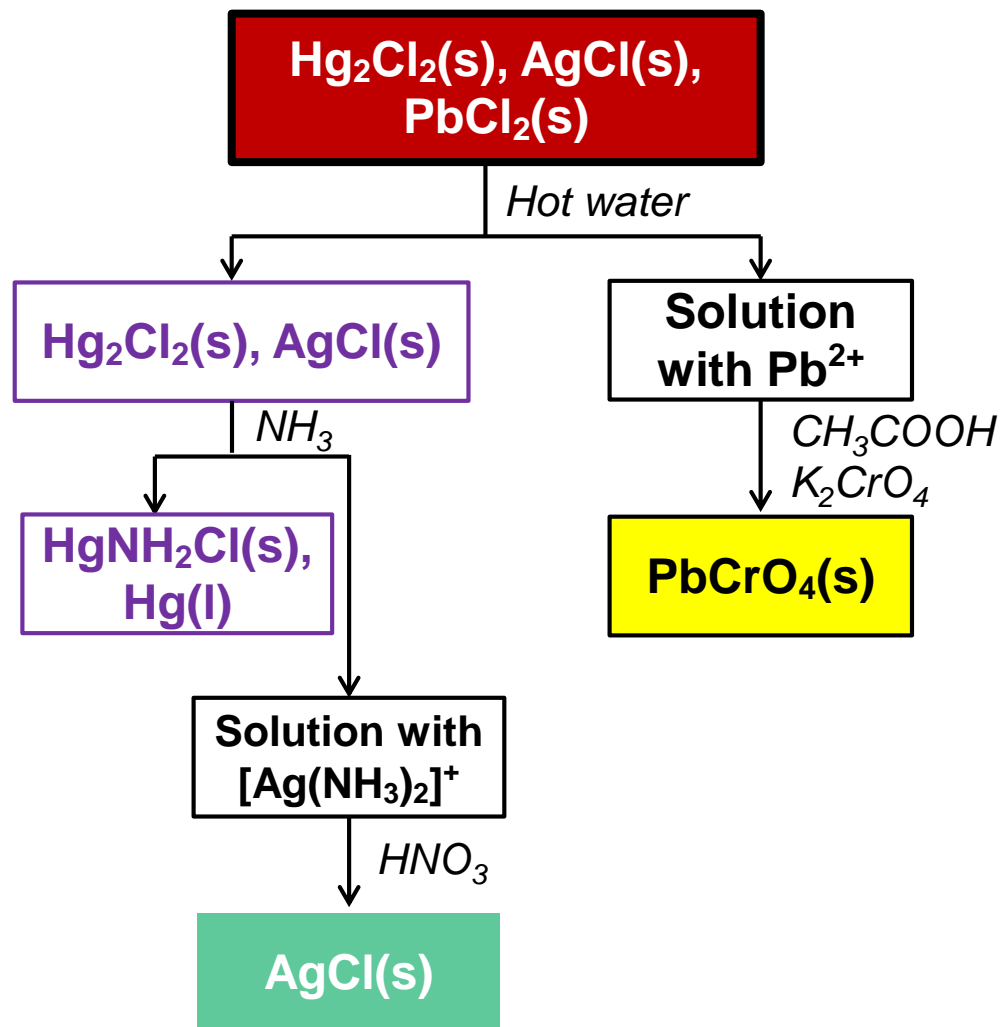
- I. Separating cations into five groups based on their solubilities in the presence of various precipitating reagents
- II. Selective and sequential dissolution of cations in the same group
- III. Verifying individual cations





# Qualitative Analysis of Cations

- I. Separating cations into five groups based on their solubilities in the presence of various precipitating reagents
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- III. Verifying individual cations





# Step 1/4: Precipitating Chlorides

- Label a centrifuge tube
- Mix 2 drops of  $\text{Hg}_2^{2+}$ , 2 drops of  $\text{Ag}^+$ , and 3 drops of  $\text{Pb}^{2+}$
- Add 2 drops of 6 M  $\text{HCl}(\text{aq})$  and stir for 1-2 min (finger-flicking, glass rod, or Vortex)
- Centrifuge and decant the supernatant



- ✓ Adding excess  $\text{HCl}(\text{aq})$  may dissolve the chloride precipitates (why?)
- ✓ Clean glass rod thoroughly after each use



# Step 1/4: Precipitating Chlorides

**Solution ( $\text{Hg}_2^{2+}$ ,  $\text{Ag}^+$ ,  $\text{Pb}^{2+}$ )**



- Add 2 drops of 6 M HCl(aq) and stir for 1-2 min
- Centrifuge and decant the supernatant into another centrifuge tube



**Ppt. 1-1**  
 $\text{Hg}_2\text{Cl}_2(\text{s})$ ,  $\text{AgCl}(\text{s})$ ,  $\text{PbCl}_2(\text{s})$

- Wash with 1 d. 6 M HCl(aq) + 10 d.  $\text{H}_2\text{O}$
- Centrifuge and decant the supernatant

**Ppt. 1-1**  
 $\text{Hg}_2\text{Cl}_2(\text{s})$ ,  $\text{AgCl}(\text{s})$ ,  $\text{PbCl}_2(\text{s})$

**Soln.**

**Soln. 1-1**

- Add 1 d. 6 M HCl(aq)
- Precipitation is complete?

**No**

**Yes**

*Keep in tube*



## Step 2/4: Isolating and Identifying $\text{Pb}^{2+}$

Temp. (°C)  Chlorides	Solubility (g/100 g $\text{H}_2\text{O}$ )				
	0	10	20	50	100
$\text{PbCl}_2$	0.67	0.80	0.97	1.64	3.23
$\text{AgCl}$	$0.7 \cdot 10^{-3}$	$1.1 \cdot 10^{-3}$	$1.6 \cdot 10^{-3}$	$5.4 \cdot 10^{-3}$	$21 \cdot 10^{-3}$
$\text{Hg}_2\text{Cl}_2$	$1.4 \cdot 10^{-3}$	$1.7 \cdot 10^{-3}$	$2.4 \cdot 10^{-3}$		





# Step 2/4: Isolating and Identifying $\text{Pb}^{2+}$

Ppt. 1-1  
 $\text{Hg}_2\text{Cl}_2(\text{s}), \text{AgCl}(\text{s}), \text{PbCl}_2(\text{s})$



- Add 5 drops DI water
- Heat in boiling water bath for 3 minutes in fume hood
- Centrifuge as quickly as possible (decant to a test tube)
- Repeat hot water extraction 2-3 times (supernatant should be free of  $\text{Pb}^{2+}$ )

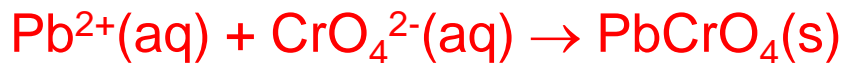
Ppt. 1-2  
 $\text{Hg}_2\text{Cl}_2(\text{s}), \text{AgCl}(\text{s})$

Soln. 1-2



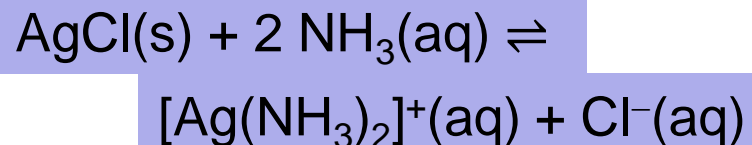
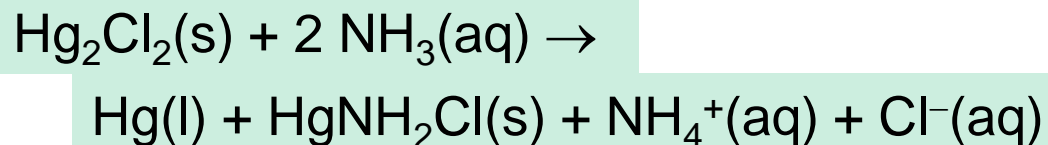
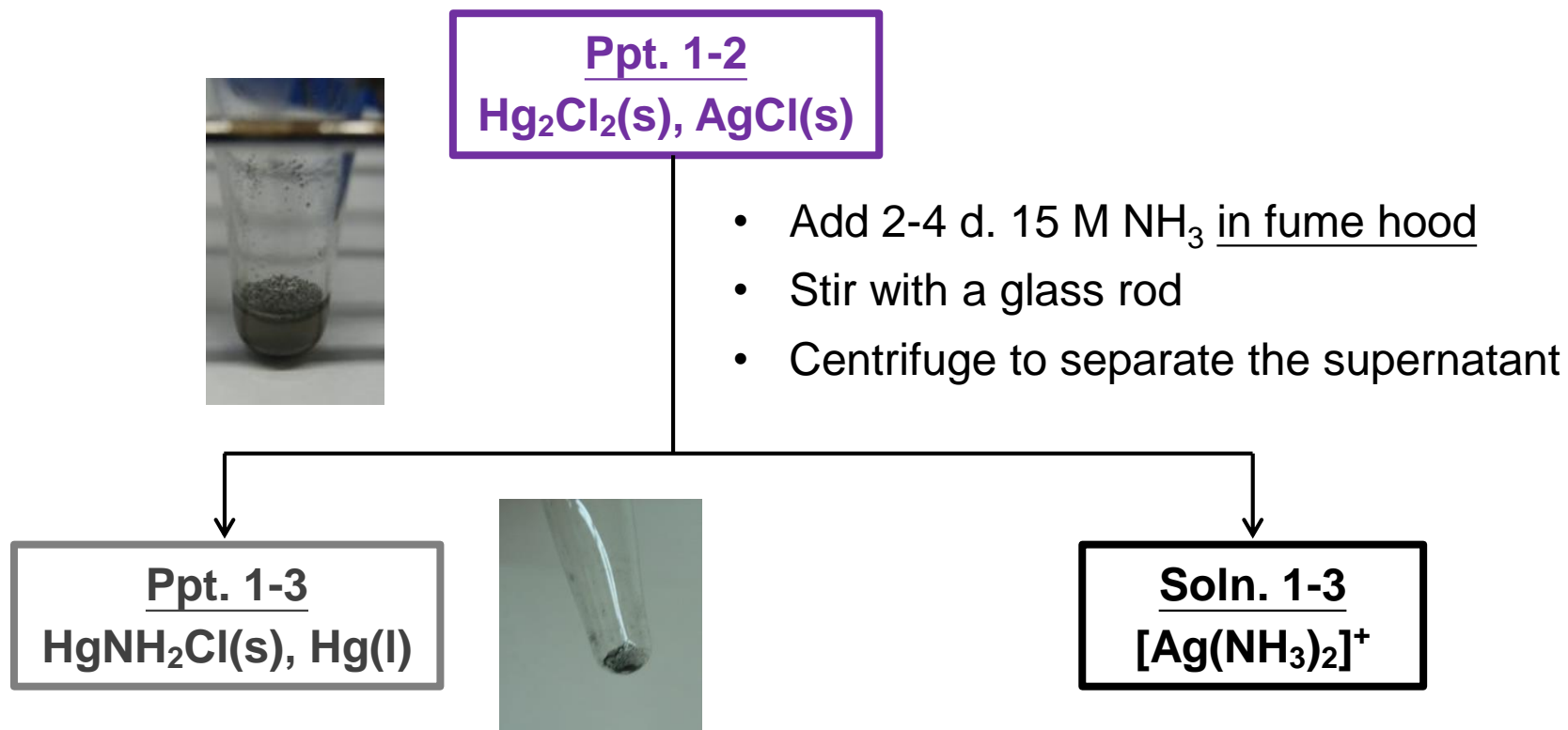
Add 1 d. 6 M  $\text{CH}_3\text{COOH}$   
& 1 d. 0.5 M  $\text{K}_2\text{CrO}_4$   
to each test tube

$\text{PbCrO}_4(\text{s})$   
(yellow ppt.)





## Step 3/4: Isolating and Identifying $\text{Hg}_2^{2+}$



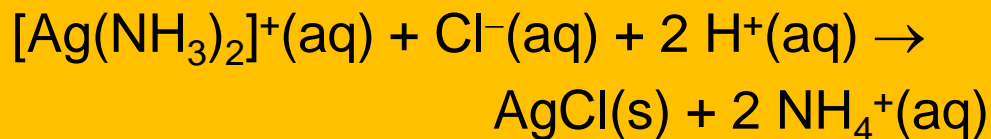
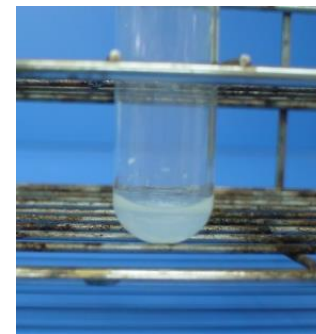


# Step 4/4: Isolating and Identifying Ag<sup>+</sup>

**Soln. 1-3**  
**[Ag(NH<sub>3</sub>)<sub>2</sub>]<sup>+</sup>**

Gradually add 6 M HNO<sub>3</sub> until the solution turns acidic (use a glass rod to spot the solution on a litmus paper)

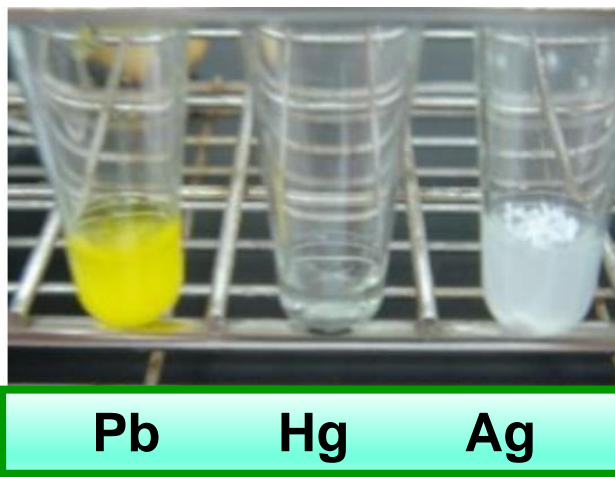
**AgCl(s)**  
**(white ppt.)**





# Record Detailed Observations

- **Operations** (e.g. adding x drops Y, centrifuge speed setting, repeating extraction z times, etc.)
- **Reaction conditions** (e.g. in fume hood, in boiling water bath, etc.)
- **Phenomena** (i.e. appearance of precipitates and solutions, speed of changes, etc.)
- Present all identification products to TA at the end of lab





# Safety Notes

- Wear NBR gloves throughout the experiment
- Hot water bath and 15 M  $\text{NH}_3(\text{aq})$  should be operated in the fume hood
- Take only the required amount of chemicals as prelab manual (minimize chemical waste)
- Use test tube rack (or test tube tong) for transporting test tubes and centrifuge tubes
- All wastes should be disposed into the heavy metal recycling container



# Clean-Up and Check-Out

- Clean and return the centrifuge tubes (sticky labels should be removed)
- Tuck the lab stools underneath the lab bench
- Clean up the lab bench and check personal equipment inventory (have an associate TA signed the check list)
- Present all identification products to TA during check-out
- This is a **Brief Report** experiment:
  - **Hand in prelab/lab note/report together to the TA**
- Groups on duty shall stay and help clean up the lab



# Centrifugation

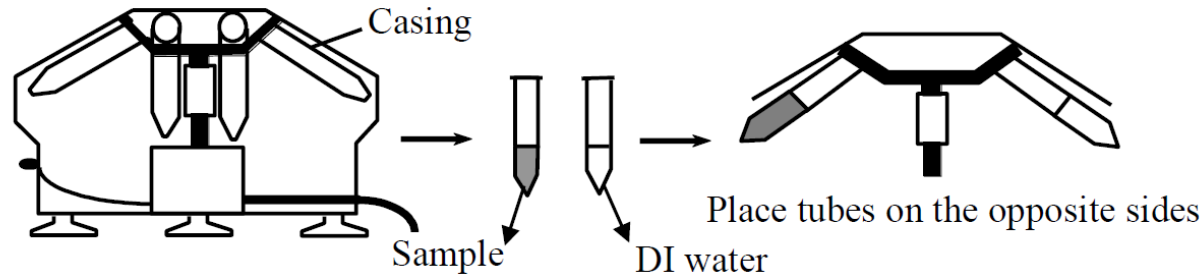


Figure T8-1 Section view of a centrifuge

[T8 Video \(YouTube link\)](#)

- Inspect whether the casings are still intact; clean or replace the casings if necessary
- Do NOT use regular test tubes in centrifuge; use only designated centrifuge tubes
- Mind the balance of the setup; only work with even number of tubes at the same time, and place them directly opposite to each other
- If only one sample solution needs to be centrifuged, take another tube with a similar amount of DI water and place it at the counterbalancing position
- Close the cover before starting the motor. Start the motor from low speed, then ramp up the speed if no malfunction is detected
- In cases of unusual sound or vibration occurring, turn the centrifuge off immediately
- Allow the rotating assembly to deaccelerate on its own after the process. Do NOT attempt to stop it manually, and open the cover only after the assembly has stopped



# Decantation

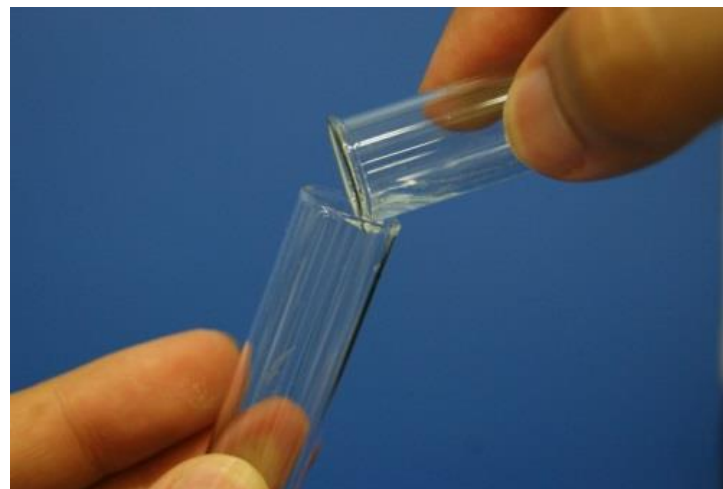
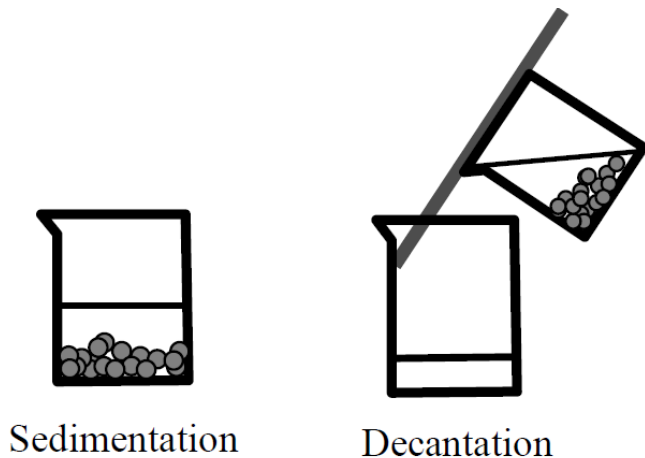


Figure T5-1 Sedimentation and decantation

*Decantation between two centrifuge tubes*

Decantation is a simple method for separating solid from liquid. The solid precipitate settles to the bottom if its specific gravity is greater than that of liquid. While there may still be some solid remaining suspended in the liquid, the separation can be achieved by carefully pouring the liquid off:

- Let the solid settle to the bottom of the mixture (or use a centrifuge – see T8)
- Pour the liquid out of beaker and use a glass rod to guide the liquid flow (Figure T5-1). This must be done slowly so that no solid is carried over.