The Preparation of PbI₂ from Galena

The preparation of lead iodide from galena (lead sulfide) has several advantages. It illustrates the preparation of a compound from a common mineral; the procedure gives a high yield; a good deal of chemistry of quite simple reactions is involved; techniques such as liquid transfer, filtration, recrystallization, and so on are used; the entire process takes up the time available in a 3-hr laboratory period. Yields have been about 90%. The following is the procedure performed by our students with questions they are expected to answer.

Procedure
All steps through #6 must be done in the hood!
1) Powder some galena in a mortar and place a weighed sample (0.5–1.0 g) into a 250-ml Erlenmeyer flask and add 30 ml conc. HCl.
2) Heat on a tripod using a gauze, removing the flame when effervescence becomes rapid and replacing it when it slows down. Keep heating in this way until the black powder is just about gone (about 10 min) and then boil gently for about 5 more min.
3) Remove the flame and carefully add 45 ml conc. HNO₃. When effervescence diminishes, heat to boiling.
4) Boil until the vapors in the flask are colorless and the liquid is a very pale yellow; then boil for 2 more minutes.
5) Transfer to a 500-ml Erlenmeyer flask using 30 ml H₂O as transfer liquid and cool the contents of the flask.
6) Add conc. NH₃ until a persistent voluminous white precipitate is obtained and then add dilute HNO₃ until the precipitate dissolves completely to give a clear solution.
7) Transfer (filter if necessary) to a 1-l Erlenmeyer flask using 200 ml H₂O as transfer and washing liquid.
8) Add a solution of 3 g KI in 20 ml H₂O.
9) Heat to boiling (careful; bumping!) with agitation until the PbI₂ has dissolved completely.
10) Turn the flame off and allow recrystallization to occur as the solution cools spontaneously.
11) When at room temperature, cool the flask under running water and then collect the precipitate on fast filter paper. Wash well with water and dry in an oven at 110–115 °C for at least 15 min.
12) Weigh the precipitate and calculate the percent yield of PbI₂ assuming the galena to be 100% PbS.

Questions
1) PbCl₂ is a white compound insoluble in water; yet in Step 2, where PbCl₂ is forming (PbS + 2HCl → PbCl₂ + H₂S) no precipitate is obtained. Why not?
2) What is the precipitate observed in Step 3 (when the nitric acid is added)?
3) In Step 4, a solution of Pb(NO₃)₂ is the product. What happened to all of the chloride ion from the HCl added in Step 1?
4) Write the equations for the formation and dissolution of the precipitate in Step 6.
5) Why is an excess of KI in Step 8 inadvisable?
6) Why are the contents of the flask not cooled down immediately under running water in Step 5 instead of waiting for the solution to cool spontaneously to room temperature?

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