Americium from urine sample

Determination of Am from urine sample

1. Take 200 – 3000 ml of urine in decanter (figure 1)

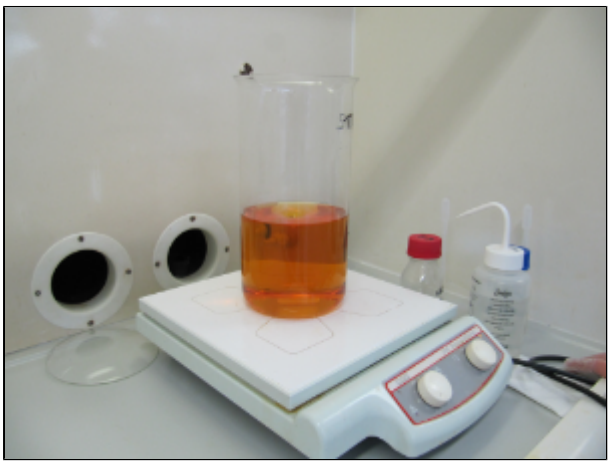

Figure 1. Urine sample in decanter

2. Add tracer, Am-243, typically between 0.02 - 0.05 Bq.
3. Put the decanter onto the double-boiler or heating plate.
4. Add 2 - 3 drops octanol and 1 ml 1.25M $Ca(NO_3)_2$. The urine need to mixed using magnetic stirrer. If you are analysing blank samples, then add 5 ml 1.25 M $Ca(NO_3)_2$.
5. Add 65 % HNO_3. The amount of acid is 10% of the sample volume e.g. 100ml of HNO_3 is added for one liter sample.
6. Heat the sample in double-boiler at 90 °C for 2 - 3 hours. Cover the sample duting heating.
7. Take sample off the boiler.
8. Add 0.2 ml 3.2 M $(NH_4)_2PO_4$ into the warm solution mixing all time.
9. Mix the sample all along using magnetic stirrer, add 25 % NH_4OH until the pH of the solution is around 9 and $Ca_3(PO_4)_2$ precipitation is composed (video 1, figures 2 and 3).

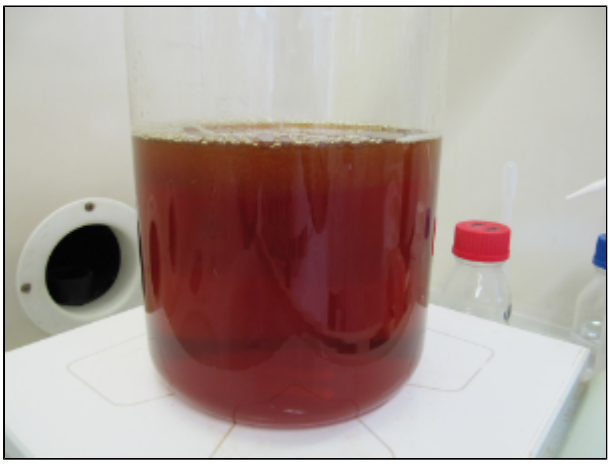

Figure 2. Formation of precipitation, at the beginning.

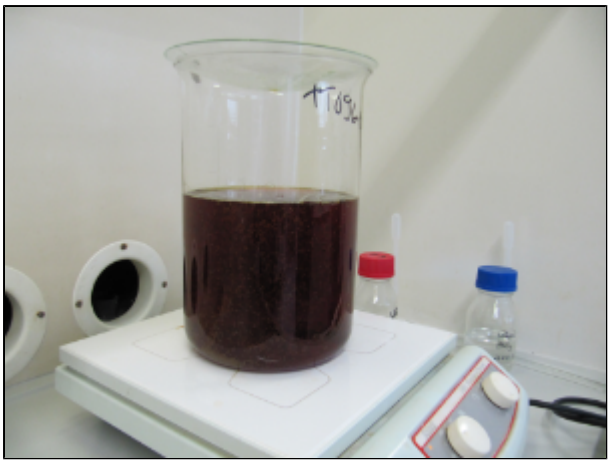

Figure 3. Ca3(PO4)2 precipitation at the end.

10. Remove the magnetic stirrer.
11. Heat the sample about 30 minutes. Turn off the heating and let the precipiation to settle down at least two hours, gladly over the night (figure 4).

Figure 4. Precipitation settling down.

12. Decant the solution. Abondon the solution (video 2).
13. Move the precipitation and remaining solution into the centrifuge tubes (figure 5). Centrifuge samples 10 minutes using 2000 rpm.

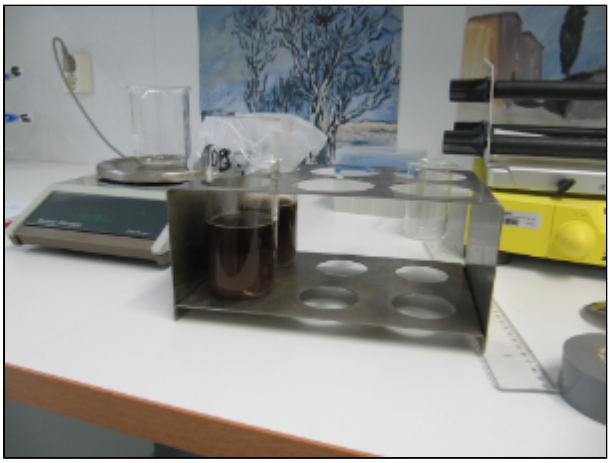

Figure 5. Sample in the centrifuge tube.

14. Move the solution, wash the the precipitation using distilled water and centrifuge samples again.
15. Abondon the solution (video 3, figures 6 and 7.).

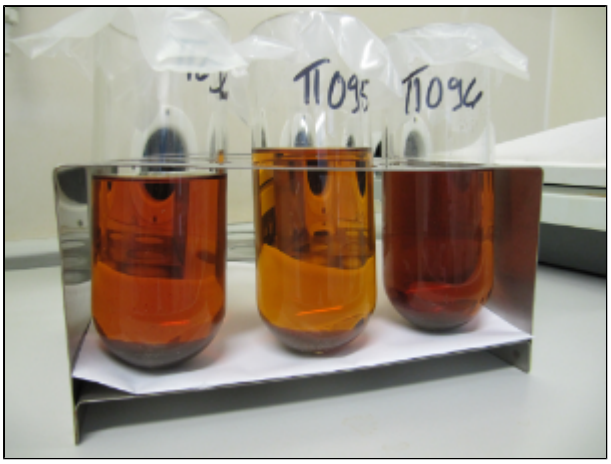

Figure 6. Samples after centrifugation

Video 3. Removing the liquid from the sample.

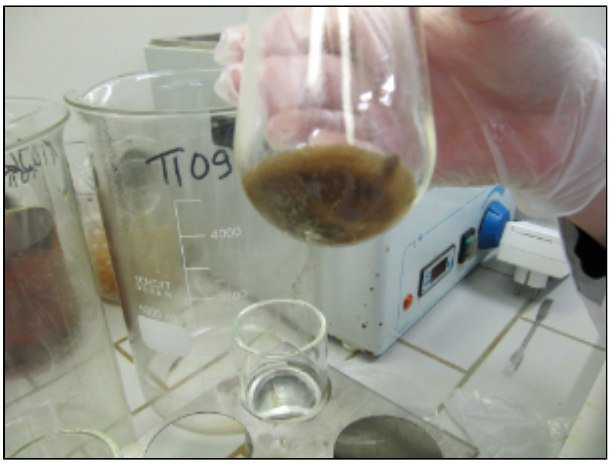

Figure 7. Precipitation.

16. Dissolve the precipitation into 5 ml conc. HNO3 and remove the solution into the 100 ml decanter. Wash the centrifuge tubes using 2 x 5 ml conc. HNO3. Add the washing solutions into the original solutions.
17 Wash the decanter using 5 ml conc. HNO3 and put washing solutions together with original solution.
18. Add 1 ml 30 % H2O2. Evaporate the solution into dryness using heating plate (150 - 200 °C). Add 2 - 3 drops 30 % H2O2 into the warm sample. This oxidate the remaining organic matter.
19. Cool the sample, add 5 ml H2O2 and evaporate the sample into dryness.
20. Cool the sample, add 5 ml HNO3 and evaporate the sample into dryness.
21. Repeat the steps 19 and 20 until the precipitate is white (figure 7.)

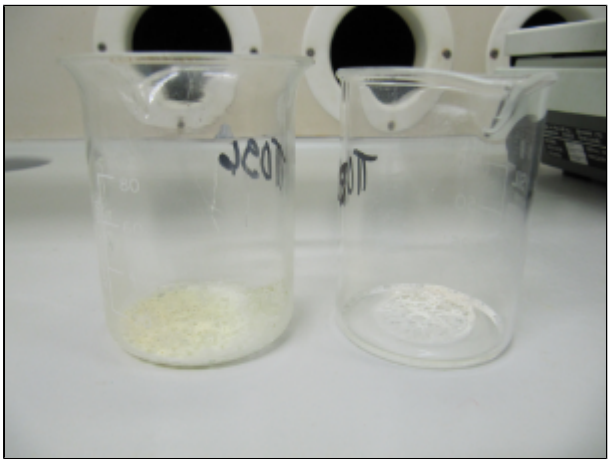
Figure 7. Not yet ready precipitation on the left and final white precipitation on the right.

22. Dissolve the residual into 20 ml 3M HNO3 - 1.0M Al/NO3)3 solution.
23. Prepare the TRU column. Put 2ml TRU resin into the column.
24. Let 5ml 2M HNO3 acid to run through the column (conditioning)
25. Load the sample into the column (figure 8). This takes about 1,5 hours.

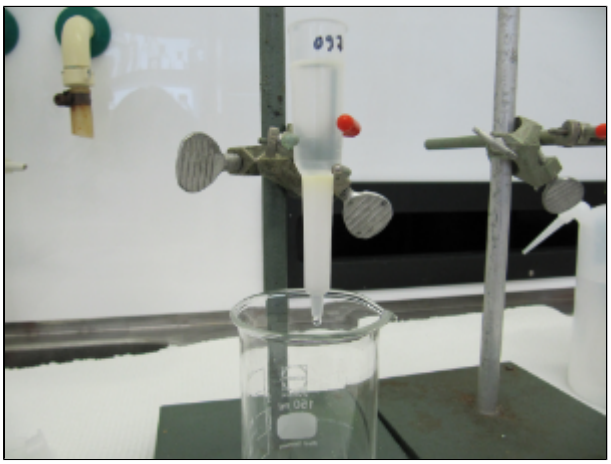

Figure 8. Separation of Am using TRU column. See the yellow colour at the top of the column.

26. Rinse the decanter and put the solution into the TRU column.
27. Add 5ml 0.5M HNO3.
28. Take a new decanter under the column and add 3ml 9M HCl into the column. Collect all solutions into the decanter.
29. Add 20ml 4M HCl into the column. Collect solutions into the same decanter.
30. Evaporate all solutions nearly dry, takrs about 1,5 hours.
31. Add carefully 3ml conc. HCl and 1ml conc. HNO3. Evaporate.
32. Repeat the step 31 twice.
33. Add 2ml conc. HCl and evaporate nearly dry.
34. Add 2ml conc. HCl and heat the decanter for 2 minutes.
35. Dilute the solution using 8ml of ultra pure water and heat the sample for 2 minutes.
36. Remove the sample into the 50ml plastic centrifuge tube.
37. Rinse the decanter using 2ml of ultra pure water and add the water into the 50ml plastic centrifuge tube.