Test 86 summary

Overall plan

This test sought to recover the zinc by initially moving it into solution with sulphuric acid, and then precipitating it as zinc hydroxide. The latter involved a two-stage precipitation, with the first precipitation aimed at removing most of the silicon.

stage 1	50g of combined dry sample in 500 ml of 5% H_2SO_4 (25 ml of 98% acid, - or 45 g of 98% acid) digested over 10 days. Then solution filtered off.
stage 2	Solution partially neutralised to pH 3.3 using 10% NaOH. Resulting precipitate filtered off. Precipitate intended for tailings.
stage 3	Remaining solution neutralised to pH 6.6 using 10% NaOH. Resulting precipitate intended to contain most of the zinc.
stage 4	Remaining solution neutralised to pH 11 with 10% NaOH. This was a check to confirm that most zinc had been removed in previous precipitate.

Overall results

While the digestion appeared to go well (estimated over 90% Zn recovery to solution), a significant percentage of the zinc (about 50%) appeared to "get lost" somehow with stage 2. Stage 3 balanced out well based on remaining zinc. Stage 4 confirmed that nearly all the stage 2 zinc had been removed at stage 3.

However, acid usage was very high, at 90% of dry ore weight. The amount of NaOH needed to precipitate the zinc was also very high, at about half the dry ore weight.

Later tests (tests 87, 89 and 90) did indicate that a lower acid concentration could be used, but indicated that it would probably not be less than about 60% of dry ore weight. This approach to the zinc recovery was abandoned in further tests.

Test procedure details

Acid digestion

50g of pulverised combined dry sample (@ an estimated 7% Zn) was stirred into 500 ml of 5% H_2SO_4 (i.e. 25 ml of 98% acid, - or 45 g of 98% acid).

A hotplate-magnetic-stirrer was used to maintain the temperature at approximately 30^0C. The hotplate-stirrer was operated only during the day, and was turned off during the evenings (I did not trust the hotplate-stirrer, - with good reason as it later turned out).

Temperatures during the night were not monitored, but would have been generally below 15^0C.

For two hours prior to sampling (during the afternoons) the hotplate-stirrer was turned off to allow the sediment to settle.

After 6 days the sample was removed from the hotplate-stirrer, and stirring was only occasional by hand-held stirring rod. Analysis was continued for a further 4 days.

Zinc recovery to solution appeared to be about 90% after 6 days, - rising, eventually, to above 95% after 10 days.

The Zinc acid solution from the above digestion (after 10 days) was recovered by filtration. This amounted to 275 ml of solution which analysed at 1.2% Zn, implying a weight of 3.3g of Zn in solution. Considering that some Zn has been lost in assays over the 10 days of the above digestion, I think the head assay, assumed from a previous analysis on the pulverised sample, was a bit lower than the sample used here.

The 275 ml of solution was neutralised in 3 steps.

First stage neutralisation

The 275 ml of solution (@ 1.2% Zn) was neutralised to pH 3.3 using 10% NaOH. The neutralisation was done by pipette, while stirring (magnetic stirrer), using 2 ml alkali intervals and then, after pH 2 using 1 ml alkaili intervals.

At the completion of the first neutralisation, the solution contained a light brown precipitate. Total volume after the neutralisation was 419 ml. After filtration 260 ml of light brown solution was obtained. The other 159 ml remained as precipitate, plus solution trapped in the precipitate.

The resulting solution analysed at 0.37% Zn
The washed precipitate weighed 2.25g @ 0.71% Zn (0.016g Zn)

Unfortunately the water from washing the precipitate was not analysed.
But if we assume that there was about 415 ml of solution (including that lost in the washing), the solution analysis implies a total of 1.5g of Zn in the solution.

There is something very wrong here. This is only half the amount of Zn there should be.
What has happened to the rest?

Second stage neutralisation

250 ml of the above solution (@ 0.37% Zn) was then used for the second stage neutralisation to pH 6.6. This produced a light blue/green precipitate, which was filtered off, leaving 190 ml of very clear colourless solution.

The resulting solution analysed at 0.014% Zn. (0.027g Zn for the 190 ml)
The washed precipitate weighed 2.85g @ 35.1% Zn (1.0g Zn)
The wash water from the precipitate (160 ml) gave 92 ppm (0.015g Zn)

The weight of the wet precipitate was 55g, which dried out to 2.85g, so the volumes are pretty well accounted for (allowing perhaps 5 ml being soaked up by the filter paper).
The total Zn implied by the analyses is 1.04g. The start solution analysis implies 0.93g was available to begin with. This discrepancy is within analysis error (+/- 5%) for the precipitate and start solution.

Things balance for this stage anyway.

3rd stage neutralisation

180 ml of the solution (@ 0.014% Zn) from the previous neutralisation was then taken up to a pH of just above 11 with further NaOH. This produced a further precipitate, which was filtered off and analysed.

The resulting solution analysed at 0.0001% Zn. (negligible Zn)
The washed precipitate weighed 1.6g @ 1.6% Zn (0.026g Zn)
Precipitate wash water (1000 ml), Zn below detectable

This stage balances very well with the previous.

Sediment analysis

The sediment remaining after the acid digestion was dried and weighed, giving 42.1g. There has been a weight loss of about 7.9 g, which matches reasonably well, given weight errors in scraping the precipitate from the filter papers, with the total precipitate weight (6.7 g).

The 42.1 g of dried sediment was then placed into 100 ml of aqua regia for analysis, but a very delayed and very sudden and violent reaction (after I concluded nothing much was going to happen) spread the sample all over a safety tray in the fume cupboard. No sensible analysis was then possible on this sediment.

The unfortunate thing here is that it looks like about half a tonne of NaOH would be required for every tonne of ore. This would be too expensive.

Samples sent to Hobart
22nd January 2008

Test 95

1. ***Stage 1 solution*** after attempt to place silicon into solution at pH 2. This is the solution decanted at the completion of stage 1.
2. ***Stage 1 wash solution***. After the initial decanting the slurry was brought back to 4 litres volume and again lowered to pH 2 with H_2SO_4 (4.5 g acid needed). This is the decanted solution after stirring and allowing to settle overnight.
3. ***Stage 2 slurry***. This is the complete slurry from the zinc digestion stage, - 19 days after adding the acid. Solution Zn assay on 21st Jan, after heating from 11^0C to 34^0C over 2.5 hours, was 0.46% Zn.
4. ***Stage 2 solution sample*** after 9 days digestion at ambient temperature. This represents the highest recovery of Zn to solution, and is the small solution sample in the test tube. It assayed at 0.54% Zn on 12th Jan.

Test 96 slurry

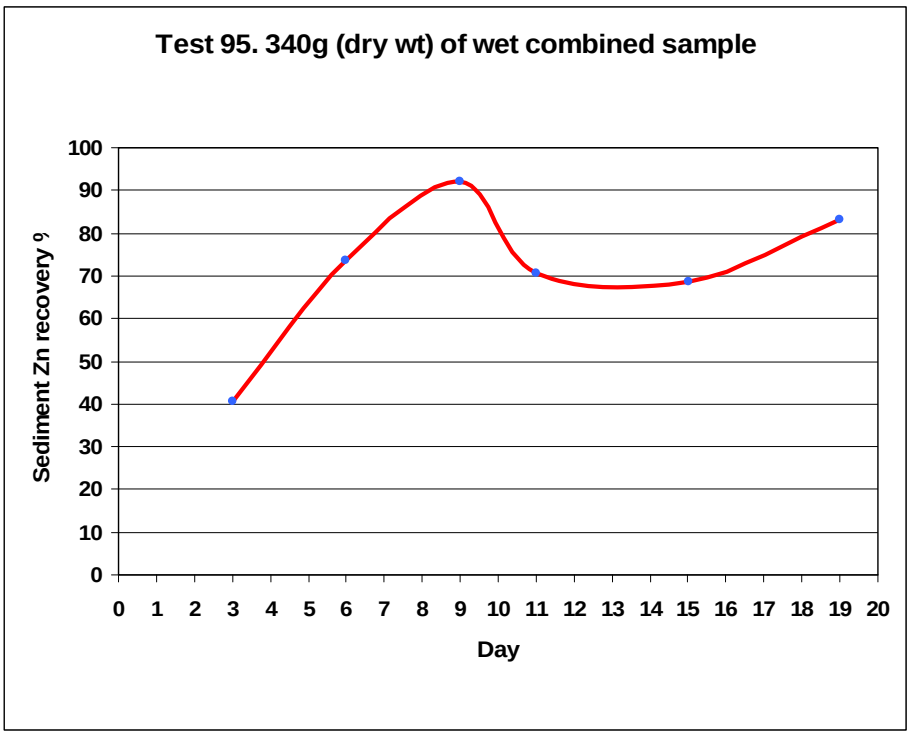
This slurry was prepared on 3rd December.
500 g wet combined sample ground in 500 ml water in small rod mill, them screened at 2 mm and originally made up to 4 litres of slurry. The volume has since been reduced by evaporation.
No acid was added. The sample was intended for a repeat of the “failed” Hobart higher temperature digestion, but this was not carried out.

Also, taped to the container, are the larger (>2mm) particles.

Further comments on Test 95

The day 6 and day 9 assays on the test 95 solution coincided with some of the hottest weather we had so far this summer. The lower two assays that followed coincided with very much cooler weather. There was therefore the suspicion that the digestion was temperature dependent, possibly due to a reversible reaction involving some remaining silicon. It had been intended to follow this up by heating and cooling the slurry and seeing if the assays followed the temperature variations.
The requirement to send the slurry to Hobart ended this idea, but some brief heating was tried today (21st January).

The slurry temperature at 9.30 am was 11^0C. At 12 midday the hotplate had increased it to 34^0C, after which the sample was allowed about an hour to settle and then sampled. The zinc assay did increase over the previous two assays, as shown in the graph.



However, I think the results of this quick test are just suggestive.

Record of test work
(January 2008)

(Tuesday 1st January to)

Test 95

500g of wet combined sample was ground in 500 ml of water. The resulting slurry was screened at 2mm and then made up to 4 litres of slurry. 10% sulphuric acid was then added to bring the pH down to 2. The dry weight of the 500g used in the test was approximately 340g, with a density of 2.1 g/cm^3 (a solids volume of 162 cm^3).

The sample was continuously stirred during the day by a rotating stainless steel paddle. The pH was monitored by periodically removing a small sample in a beaker. After measuring, the test sample was returned to the bucket.

A total of 44.1g of 98% H_2SO_4 had been added to the slurry to bring the pH down to 2 at the end of November 2007. The resulting solution was analysed for Zn, giving 600 ppm for 3838 ml of solution. This amounts to a removal of 10% to 11% of the available Zn from the ore.

This test had been ceased at this stage, in preparation for the major test designed by Bob.

When again looked at on 1st January, and after adding water to bring the slurry volume back to 4 litres (evaporation over the past month had lowered the volume), the pH had risen to 2.7. Note: the pH meter was calibrated with 0.01N HCl.

A further 9g of 98% acid was required to bring the pH back down to 2, giving a total acid usage so far of 53.1g (15.6% of dry ore weight).

The slurry was then let stand overnight before commencing to draw off the solution (hopefully containing most of the soluble silicon). This amounted to 3.2 litres of solution. The slurry was then made up again to 4 litres with water, and 25 ml of 10% acid was needed to bring the pH back to 2.02 to avoid any remaining soluble silicon from coming out of solution. After several hours stirring, this was allowed to settle overnight before again decanting the solution (approx 3.2 litres). Note that the second 25 ml addition of 10% acid has not been included in the total acid usage.

Up till this point there was no obvious smell of H_2S.

Both decanted solutions were retained for later analysis.
Initial 3.2 litres of solution gave 900 ppm Zn (indicating 2.9g Zn in the 3.2 litres)
(and 3.67g in the 4.1 litres of total solution)
Wash solution of 3.2 litres gave 230 ppm Zn (indicating 0.74g Zn in the 3.2 litres)
(and 0.93g in the 4.1 litres of total solution)

Therefore the total Zn extracted by the silicon removal is about 3.8g out of an estimated total Zn of about 25.8g, based on the test 94(b) Zn balance. This indicates approximately a 15% loss of Zn to the silicon removal.

500 ml of 10% (90g of 98%) H_2SO_4 was then added to the slurry, and stirred for about 4 hours before allowing to settle overnight. At this stage the acid useage was

Removal of silicon	53g	
Zn Digestion	90g	
Total	143g	(42% of dry ore weight)

A strong smell of H_2S was then evident.

After 3 days digestion, with constant stirring during the day and sample settling overnight, at an average of about 20^0C; -

Zn recovery (@ 0.21%) - approximately 40% of Zn remaining in sediment.
- approximately 34% of total sample Zn.

After 6 days digestion (volume reduced to 3838 ml, - due to evaporation)

Zn recovery (@ 0.42%) - approximately 73% of Zn remaining in sediment.
- approximately 62% of total sample Zn.

After 9 days digestion (volume reduced to 3638 ml, due to evaporation)

Zn recovery (@ 0.55% Zn) - approximately 91% of Zn remaining in sediment.
- approximately 78% of total sample Zn.