CHM 2990

Project Notes
(Logbook)

Michael Walton

Research conducted Dec. 2017 and Jan-Feb. 2018
in the laboratory of Dr Alison Funston

Front Note:

These notes were compiled on Nov. 25, 2022 based on the written records and files created over the course of the summer 2017-2018. This compositions is done by the author (Eliot Walton).

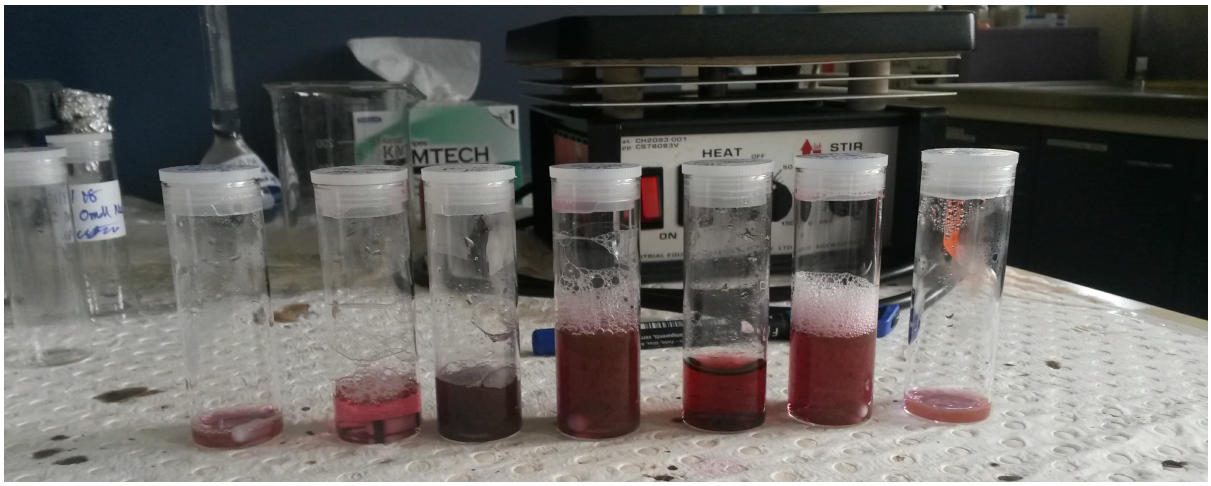


Figure 1: Photograph of gold nano-particle solutions on laboratory work bench. Taken by M. Walton, summer 2017-18.

119835

TOPIC: Formalisee method spheres.

NAME: Michael Walton. DATE: 20/12/17

1. Preclean all required glassware with Aqua Regia and heat oil bath to 100°C.

2. In round bottom flask, Mix 8.9mL of H_2O and 100µL of 0.05 M $HAuCl_4 3H_2O$. Swirl to mix solutions. Heat with constant stirring for (in 100°C bath about 10 minutes.

3. Weigh precisely 0.037 g of trisodium citrate and dissolve in 1.86 mL of H_2O. Heat and add 1mL to ~~about~~ Au Soln.

4. Stirr and heat for about 15 min Run ~~keep at~~ cool to room temperature. with stirring.

Attempt 1
- Soln failed to produce particles - turned blue then light purple - Run placed inside of flask. Na-Cit add incorrectly. Amend method to 1mL of Na-cit

Attempt 2.
- Thermometer indicates temp ~80°C, took much longer for light purple -> dark purple -> red (~10 min longer). Spheres thought to be small.

Attempt 3.
- Thermometer indicates temp ~100°C, transition from light -> dark purple -> red occurred @ ~4 min -> 8 min -> 13 min. Red a deep ruby colour - good sized spheres.

Formalised Method of Spheres:

1. Pre-clean all required glassware with Aqua Regia while heating oil bath to 100^{o}C.

2. In a round bottom flask, mix 8.9mL of H_2O and 100μL of 0.05M of $HAuCl_4.3H_2O$ swirl to mix solutions. Heat with constant stirring in 100^{o}C oil bath, for about 10 minutes.

3. Weigh precisely 0.0137g of tri-sodium citrate and dissolve in 1.86mL of H_2O heat and add 1mL to gold solution.

4. Stir and heat for about 15 min then cool to room temperature with stirring.

Attempt 1.

Solution failed to produce particles – solution turned blue then turned light purple – then plated inside of flask. Tri-sodium citrate add incorrectly and too much. Amended method to 1mL of tri-sodium citrate.

Attempt 2.

Thermometer indicates temperature at 80^{o}C, took much longer for solution to turn light purple then became dark purple then deep red, for 10 minutes of longer. Spheres formed but are suggested to be too small.

Attempt 3.

Thermometer indicates temperature at 100^{o}C, transition from light purple at 4 minutes to dark purple at 8 minutes then ruby red at 13 minutes. The ruby red indicates that the spheres are of a good size.

Vials in to dry 11:30am
15/1/18.

2.3450 g

0.2M

$c = \frac{n}{L}$ G.1935

$V = n/c$

0.006435

$M = \frac{wt}{Mol.wt} \times \frac{1000}{V(ml)}$

$\Rightarrow 0.2 = \frac{2.3450}{364.46} \times \frac{1000}{V}$

$V = \frac{2.3450 \times 1000}{364.46 \times 0.2}$

2345

72.892

32.2 ML

119836

TOPIC: Lab notes

NAME: Michael Welton DATE: 15/1/18

Gold nanocube method.

Solutions:

1. 0.01 M NaBH4 10mL ~ 0.0056gm + 15mL H2O
2. 0.05 M '
3.
4.

Refer to attack pg.

Using CTAB.

- Rock gurl bottle cleaned & stir heated.
- 2.3450 g of CTAB in weighing boat.
- spatula wrapped in parafilm.
- 32.170 mL of H2O added to bottle of CTAB.
- Solution heated @ 60°C while stirred.
  - turned from milky & opaque to clear.
  - time began 1:15 pm.

Calculation.

$$c = n/V \Rightarrow c = (m/M)/V$$

$$c = m/MV$$

$$\therefore M = \frac{m}{M} \times \frac{1000}{V(mL)}$$

Given $M = 364.46$.

$\therefore V = 32.170$ mL

Ligand Exchange

- Gold NPs synthesised by Turkevich Frens method;
(0.2M) ~~CTAB formation of seed particles added to a solution of~~

- @ Au-NPs.
↓
Temp reduced to 60°C and 10mg MDDA added.
dissolved in 0.5mL of MeOH
↓
5 min to pass.
Then turn off heating & allow soln. to cool to room temperature.
(Excess MDDA precipitates out).
↳ remove w/ syringe.

Film formation.

75mL of 1,2-dichloroethane and 100mL solution — (25 mL NP, varying amounts of 1M NaCl topped up with ultra-pure H_2O) in

- Wide-mouth Soda lime glass jar 250mL (88 mm height 73 mm diameter).
↓
shake @ 10s to ~~produce~~ mild emulsification
↓
5 min approx ^4 equilibrium separation.

100% RECYCLED PAPER 100% RECYCLED PAPER 100% RECYCLED PAPER 100% RECYCLED PA

Gold Nanolayer synthesis
(2018/01/15) For Micheal

Note: Thorough mixing is required after addition of every component.

Solutions:

1) 0.01M Sodium Borohydride ($NaBH_4$) solution 10 mL: ~ 0.0056gm + 15ml MQ-water
2) 0.05M gold (III) chloride hydrate ($HAuCl_4$) Solution : Stock solution from Laurent
3) 0.2M CTAB solution ~ 2.1867gm + 30ml MQ- water
4) 0.1M Ascorbic acid ~ 0.088gm +5ml MQ-water

Synthesis of seed solution:

1. 1.875 ml 0.2M CTAB solution, 2.83ml MQ-water and 0.025 ml and $HAuCl_4$ are mixed thoroughly.
2. 0.3ml $NaBH_4$ solution is then added into the above mixture with vigorous stirring for 10 mins.
3. The resulting seed solution is kept in water bath for 40 mins before use.

Growth of Nanocubes:

1. The seed solution is diluted 10 times with MQ-water for further use.
2. 1ml 0.2 M CTAB solution and 0.1ml $HAuCl_4$ solution are added into 11.38 ml MQ-water. The mixture is thoroughly stirred and kept still at least 5 min for homogenization.
3. 1.5ml Ascorbic acid is then added. (Mix thoroughly and wait for 5 min)
4. Finally, 0.025ml diluted seed solution is then added. (Mix thoroughly)
5. The final solution is then kept in water bath.

Ligand Exchange:

1. To a solution of Au-NPs, at a temperature of 60^{o}C, add 10mg of MDDA, which is dissolved in 0.5mL of MeOH.
2. Wait 5 minutes for the exchange to occur, them turn off heating and allow solution to cool to room temperature: excess MDDA precipitates out and the remove with syringe.

Film Formation:

1. In a wide-mouth soda lime glass jar, (250mL – 88mm high and 73mm in diameter) or equivalent vessel, add 75mL of 1,2-dichloroethane and 100mL solution, which contains 25mL of NPs, varying amounts of 1M NaCl and topped up to 100mL with ultra-pure water.
2. Shake for 10 seconds to promote emulsification.
3. Allow, approximately, 5 minutes for equilibrium separation.

Preparing Gold Nanocubes:

Protocol: **Gold Nanocube synthesis**
(2018/01/15) For Michael

Note: Thorough mixing is required after addition of every component.

Solutions:

5) 0.01M Sodium Borohydride ($NaBH_4$) solution 10 mL: ~ 0.0056gm + 15ml MQ-water *[needs to be made fresh every time – degrades over time]*
6) 0.05M gold (III) chloride hydrate ($HAuCl_4$) Solution : Stock solution from Laurent
7) 0.2M CTAB solution ~ 2.1867gm + 30ml MQ- water
   [does not degrade, can use a single stock solution]
8) 0.1M Ascorbic acid ~ 0.088gm +5ml MQ-water
   [needs to be made fresh every time]

Synthesis of seed solution:

4. 1.875 ml 0.2M CTAB solution, 2.83ml MQ-water and 0.025 ml and $HAuCl_4$ are mixed thoroughly. [use pippet -> volumes, 1875, 2830 and 25 uL]
5. 0.3ml $NaBH_4$ solution is then added into the above mixture with vigorous stirring for 10 mins. [" " volume, 300 uL]
6. The resulting seed solution is kept in water bath for 40 mins before use.
   [seeds also must be used the same day as made; they degrade over time]
   -Look for light brownish/yellow colour at this stage, indicates good seeds. If colour not observed remake seeds – unlikely to grow cubes otherwise.

Growth of Nanocubes:

6. The seed solution is diluted 10 times with MQ-water for further use.
7. 1ml 0.2 M CTAB solution and 0.1ml $HAuCl_4$ solution are added into 11.38 ml MQ-water. The mixture is thoroughly stirred and kept still at least 5 min for homogenization.
8. 1.5ml Ascorbic acid is then added. (Mix thoroughly and wait for 5 min)

9. Finally, 0.025ml diluted seed solution is then added. (Mix thoroughly)
10. The final solution is then kept in water bath.
    - Look for purplish / light pink colour, indicates cubes have been grown.

Notes on CTAB preparation:

- Stock bottle (Schott bottle) cleaned along with stir bead for storage of the solution.
- 2.3450g of CTAB weighted out in a weighing tray, transferred by spatula wrapped in parafilm.
- 32.170mL of H_2O added, amount in method amended to compensate for excess CTAB.
- Solution heated at 60^oC while turned
  -began at 1:15pm
  -taken off at 3:15pm

Calculation of amended volume:

Molarity = (m/M)*(1000/V_{mL})

m = 2.3450g
M = 364.46 g/mol
Molarity = 0.2M

⇨ V = 32.170mL

Observations Seed Soln. 16/1/15.

Solution bubbles vigorously - foamy bubbles.

- Auric chloride yellow lustrous solution.

- turned solution similar colour when added.

- Solution turned a light brown - yellowish colour after $NaBH_4$ add also see evolution of bubbles.

Put in H_2O Bath @ 10:33 am

Return 11:03 am.

11:43 am in.

Solution turned from orange/yellow to colourless almost instantaneously upon addition of ascorbic acid.

11:48 am in

@12:50 pm Solution now dark purple - perhaps showing growth of gold nanocubes !!!

$0.00644\ g$

$M = {}^{m}/_{M} \times \frac{1000}{V_{mL}}$

$\frac{0.01 \times 32.17}{}$

$V_{(mL)} = \frac{0.0064}{37.83} \times \frac{1000}{0.01}$

$0.000169 \times$

$= 16.9\ mL$

$= 16900\ \mu L$

$NaBH_4$ added 10:23 am

$C_6H_8O_6$

$6 \times 12 + 8 \times 1 + 6 \times 16 \Rightarrow 176$

$0.0911\ g.$

$V_{(mL)} = {}^{m}/_{M} \times \frac{1000}{Mc}$

$= \frac{0.0911}{176} \times \frac{1000}{0.1}$

$= 0.000517 \times 10000$

$= 5.176\ mL.$

4000 T 1176

- Solution turned dark yellow - almost orange when Au-Cl was added to CTAB & H_2O in 2 of Nano-Au Growth.

119837

TOPIC: Nano Cube Synthesis (1)

NAME: Michael Walton DATE: 16/1/18

Method: - as per Simon's Protocol

Amendments: 1. 0.0064 g of $NaBH_4$ used not 0.0056 g.

$V_{mL} = \frac{mass}{mw} \times \frac{1000}{[c]}$

Solved as required

2. 0.0911 g of $C_6H_8O_6$ (AA acid) used not 0.0888 g.

3. H_2O amounts adjusted

AA. $V(H_2O) = 5.176$ mL | $NaBH_4$ $V(H_2O) = 16.9$ mL

Observations: Seed solution

- Auric chloride ($AuCl_4 \cdot 3H_2O$) turned the solution of CTAB & H_2O lustrous light yellow.
- Solution turned a light brown / yellowish colour upon addition of $NaBH_4$. Bubbles - foamy - also evolved upon $NaBH_4$ addition.

Put in H_2O Bath at 10.33 am return 11:03 am

Note - Stirring increased at $NaBH_4$ addition - added at 10:23 am.

Calculation

$V_{mL} = \frac{mass}{mw} \times \frac{1000}{[c]}$

$mass = 0.0064$ g

$mw = 37.83$ g mol^{-1}

$[c] = 0.01$ M

$V(mL) = 16.9$ mL

119838

TOPIC: Nanocube Synthesis (1)

NAME: Michael Walton. DATE: 16/1/18

Method - Growth of cubes preformed in accordance with Simon's ~~Method~~ protocol.

Amendments - 1.1mL of Au-seed used diluted by 10mL of H_2O.

2. Dilution was not done until ascorbic acid was added to cube solution.

3. 0.0911g of $C_6H_8O_6$ (Ascid) was used not 0.088g.

AA. (Volume) = 5.176 mL

Observation & Growth of Nanocubes:

- Once $AuCl_4 \cdot 3H_2O$ was added to CTAB & H_2O solution turned dark yellow - nearly orange in colour.

11:43 am - ascorbic acid added.

- Solution turned colourless & transparent almost as soon as A.acid was added - no ~~measurable~~ noticable delay between addition and colour change.

11:48 am - Seeds added.

- Solution stirred vigorously before a tinge of light pink seen. Over next 5min solution slowly became light pink.

11:55 - Soln added to H_2O Bath.

Nanocube synthesis (I) – seed solution preparation (16/1/18)

Method: As per protocol, reproduced below.

Synthesis of seed solution:

7. 1.875 ml 0.2M CTAB solution, 2.83ml MQ-water and 0.025 ml and $HAuCl_4$ are mixed thoroughly.
8. 0.3ml $NaBH_4$ solution is then added into the above mixture with vigorous stirring for 10 mins.
9. The resulting seed solution is kept in water bath for 40 mins before use.

Amendments:

I. 0.0064g of $NaBH_4$ used, not 0.0056g, volume amended as per calculation at the end of these notes.
II. Stirring needs to be increased, to near maximum, when $NaBH_4$ is added.

Observations: Seed Solution preparation.

- The addition of Auric Chloride ($AuCl_4.3H_2O$) turned solution of CTAB and H_2O a light lustrous yellow, similar in colour to that of the stock solution of $AuCl_4.3H_2O$.

- Solution turned a light brown/yellowish colour upon the addition of $NaBH_4$. Additionally, a thin foam of bubbles evolved upon $NaBH_4$ addition.

Solution added to H_2o bath at 10:33am, left for ½ an hour, returned at 11:03am.

Calculation of amended volume:

$V_{mL} = (m/M)*(1000/[C])$

m = 0.0064g
M = 37.83 $gmol^{-1}$
[C] = 0.01M

⇨ V = 16.9mL

Nanocube synthesis (I) – Growth of Nanocubes: (16/1/18)

Method: As per protocol, reproduced below.

Growth of Nanocubes:

11. The seed solution is diluted 10 times with MQ-water for further use.

12. 1ml 0.2 M CTAB solution and 0.1ml $HAuCl_4$ solution are added into 11.38 ml MQ-water. The mixture is thoroughly stirred and kept still at least 5 min for homogenization.

13. 1.5ml Ascorbic acid is then added. (Mix thoroughly and wait for 5 min)

14. Finally, 0.025ml diluted seed solution is then added. (Mix thoroughly)

15. The final solution is then kept in water bath.

Amendments:

I. 1mL of Au-seed solution used and then diluted by 10mL of H_2O.

II. Dilution was not done until ascorbic acid (AAcid) was added to the cube solution. Essentially, step 1 was performed at step 4, pitot to the addition of diluted seed addition. Increased rate of stirring at that point.

III. 0.0911g of (AAcid) $C_6H_8O_6$ added not 0.088g. Volume amended as per calculation at the end of these notes.

IV. The seeds should be added to the solution without delay at the appropriate step.

V. Cubes were removed from the H_2O bath, observed, and replaced one hour after the completion of the synthesis. Recommended that cubes be left overnight to grow before being removed from the H_2O bath.

Observations: Growth of Nanocubes:

- Once Auric Chloride ($AuCl_4.3H_2O$) was added CTAB and H_2O solution turned a transparent dark yellow, nearly orange colour. This is for step 1 of the protocol.

11:43am - AAcid added to the solution.

- Solution turned colourless and transparent with no noticeable delay upon the addition of AAcid.

11:48am - Au-seed solution added.

- Solution stirred vigorously before a tinge of light pink is observed. Over the next 5 minutes of stirring, the whole solution slowly takes on the same light pink colour.

11:55am – solution added to water bath.

12:50 pm – solution inspected for colour change.

- Solution now dark purple in colour when removed from the water bath, replaced in after quick inspection.

Calculation of amended volume:

$V_{mL} = (m/M)*(1000/[C])$

m = 0.0911g
M = 176 $gmol^{-1}$
[C] = 0.1M

⇨ V = 5.176mL

Sample Preparation: **(17/1/18)**

Method: As per protocol, reproduced below.

Cleaning of ITO and Silica Slides:

16. Wash with MQ-H_2O by putting a small volume in a previously cleaned glass vial, and sonicate for 10-20 minutes.

17. Empty waste and squirt with MQ-H_2O.

18. Wash with ethanol by putting a small volume in a previously cleaned glass vial, and sonicate for 10-20 minutes.

19. Empty waste and squirt with ethanol.

20. Wash with isopropanol by putting a small volume in a previously cleaned glass vial, and sonicate for 10-20 minutes.

21. Empty waste and squirt with isopropanol.

Amendments:

I. Sonication of all solutions preformed for 20 minutes.

Method: As per protocol, reproduced below.

Washing of Gold Nanocubes:

1. Pipette out 0.1mL of cube solution into (_), reproduce as many times as desire, but ensure that an even number of vials is used as centrifuge must be balance.

2. Use centrifuge, ensuring that all vials are place opposite another; this ensure the centrifuge is balanced and functions correctly.

3. Spin at 8,000 rpm for 20-30 minutes.

4. Remove form centrifuge, particles should have collected at the base of the vial, remove the top 0.9mL of solution and dispose of as waste.

5. Pipette a further 0.9mL of MQ-H_2O onto of anaylte.

Amendments:

I. 4 of Simon's cube sample was used, each volume appeared to be different however, due to poor pipetting technique. Though amended at the fourth step, this should be noted. These samples where spun in the centrifuge for 30 minutes.

II. 2 of own cube sample was used, each volume appeared to be different however, due to poor pipetting technique. Though amended at the fourth step, this should be noted. These samples where spun in the centrifuge for 20 minutes.

Observations:

- The 4 of Simon's cubes, once completely washed, were colourless and transparent.
- The 2 of own cubes, once completely washed, had a light red colour with a slightly pink tinge.
- Differing concentrations is thought to be the origin of the colour difference.

Nanocube synthesis (II) – Seed Solution Preparation **(18/1/18)**

Method: As per protocol, reproduced below.

Synthesis of seed solution:

10. 1.875 ml 0.2M CTAB solution, 2.83ml MQ-water and 0.025 ml and $HAuCl_4$ are mixed thoroughly.
11. 0.3ml $NaBH_4$ solution is then added into the above mixture with vigorous stirring for 10 mins.
12. The resulting seed solution is kept in water bath for 40 mins before use.

Amendments:

1. 0.0065g of $NaBH_4$ used, not 0.0056g, volume to 17.18mL amended as per calculation at the end of these notes.
2. Stirring needs to be increased, to near maximum, before $NaBH_4$ is added.

Observations: Seed Solution preparation.

- The addition of Auric Chloride (HAuCl4) turned solution of CTAB and H_2O a transparent amber colour, slightly deeper than the colour of the auric chloride solution [0.05M](?).

$NaBH_4$ added at 10:30am.

- Solution turned a light-yellow brown colour upon the addition of $NaBH_4$, stirring increased after addition and thin layer of foamy bubbles evolved on surface.

Added to H_2O bath at 10:41am.

Calculation of amended volume:

$V_{mL} = (m/M)*(1000/[C])$

m = 0.0065g
M = 37.83 $gmol^{-1}$
[C] = 0.01M

⇨ V = 17.18mL

Nanocube synthesis (II) – Nanocube Growth **(18/1/18)**

Method: As per protocol, reproduced below.

Growth of Nanocubes:

22. The seed solution is diluted 10 times with MQ-water for further use.

23. 1ml 0.2 M CTAB solution and 0.1ml $HAuCl_4$ solution are added into 11.38 ml MQ-water. The mixture is thoroughly stirred and kept still at least 5 min for homogenization.

24. 1.5ml Ascorbic acid is then added. (Mix thoroughly and wait for 5 min)

25. Finally, 0.025ml diluted seed solution is then added. (Mix thoroughly)

26. The final solution is then kept in water bath.

Amendments:

I. 1mL of Au-seed solution used and then diluted by 9mL of MQ-H_2O.

II. Dilution was not done until ascorbic acid (AAcid) was added to the cube solution. Essentially, step 1 was performed at step 4, pitot to the addition of diluted seed addition. Increased rate of stirring at that point, but not until seeds were added. *In future, rate of stirring should be increased before the addition of the seeds.*

III. 0.2254g of (AAcid) $C_6H_8O_6$ added not 0.088g. Volume amended to 14.50mL as per calculation at the end of these notes.

IV. Cubes were removed from the H_2O bath, observed, and replaced one hour after the completion of the synthesis. Recommended that cubes be left overnight to grow before being removed from the H_2O bath.

V. Two solutions made, α and β, same volume added to both solutions. α had a larger stir bit than β, other than that, no noticeable or systematic difference between the two.

Observations: Growth of Nanocubes:

11:33am - $HAuCl_4$ added.

- Once Auric Chloride ($HAuCl_4$) was added CTAB and H_2O solution turned a transparent orange-yellow colour.

11:39am – Aacid added to solution.

- Solutions turned colourless and transparent with no noticeable delay upon the addition of Aacid.

11:49am - Au-seed solution added.

- Solutions stirred vigorously before light pink tinge was observed. Over the next 5 minutes of stirring, the colour of the whole solution slowly deepens to a rich, dark purple.

11:54am – solutions added to water bath.

1:42 pm – solution inspected for colour change.

- Solutions now dark purple in colour when removed from the water bath, replaced in after quick inspection.

Calculation of amended volume:

$V_{mL} = (m/M)*(1000/[C])$

m = 0.2254g
M = 176.12 $gmol^{-1}$
[C] = 0.1M

⇨ V = 14.50mL

119842

TOPIC: Lab notes

NAME: Michael Walter DATE: 2/21/18

- 11:15 am - Minispin ependorf
- ependorfs of α + β (VIII - XII) washed.

Method for Concentrated Washing:

1. Pipette 1.5mL of reaction soln into ependorfs.
2. Centrifuge @ 8000 rpm for 10 min.
3. Remove supernatant leaving pellet.
4. Pipette 1.5mL of reaction solution into same ependorfs (on top of pellets).
5. Centrifuge @ 8000 rpm for 10 minutes.
6. Remove Supernatant leaving pellet. (leave 100μl)
7. Make up to ependorf 1.5mL mark w/ MQ-H2O (glass 1mL pipette recommended).

- Final soln may have some residual pellet at bottom — recommend 10 sec vortex and/or 15-20 second sonication.
- Deposited in clean vial ~~above~~ by pipette
- ~~200μ~~ 200μL added to 3.5mL in cuvet.

Note on [Au]; Abs = 0.3 ⇒ M = 1.25×10^{-4} M

119843

TOPIC: Synthesis Au - III 3 -

NAME: Michael Crutton DATE: 23/1/18

Method - as per protocol w/ the following key amendments

Amendments:

1. 0.0106g of $NaBH_4$ used.
2. Ice cold (chilled) $NaBH_4$ added - Ice filled ~5/6 of a 250 mL beaker - $NaBH_4$ soln 'buried' in ice. Done for 18 min
3. Stirring increased before addition of seeds.

Observations:

- $NaBH_4$ added to ice bath 10:36 am
  - So small bubbles evolved in solution
- $HAuCl_4$ added to seeds @ 10:48 am
  - Solution turned transparent red amber.
- $NaBH_4$ added @ 10:54 am
  - Solution turned light brown/yellow upon addition of $NaBH_4$. (Stirring increased.
- Au - Seeds ~~added~~ in Water bath @ 11:05 am - temp 30°C ± 1°C

119848

TOPIC: Lab notes

NAME: Michael Walton. DATE: 25/11/15

UV-vis Sample - 2000μL H_2O
+ 500 μL sample.
Blank - 2500μL.

Ligand:

- Soln removed from stirring @ 2:33pm cloudy reddish soln. w/ a hinge of purple.
- ~ 1.1mL soln. pipetted into 2x eppendorf centrifuged for 10 min @ 8000 rpm.
- Red pellet observed at bottom when removed from centrifuge colourless supernatant removed, leaving 100μL of soln. & pellet.
- Redispersed into 1.4mL of ligand soln. Centrifuged twice (both 10min @ 8000 rpm) as supernatant showed a hint of colour. Still shows a hint of colour after 2nd centrifuge.
- Supernatant removed and 3rd wash done @ 10min at for 8000 rpm and redispersed in ~~Ligand~~ ~~Water~~ ~~Soln~~. pH tested to be 5, adjusted to 8 with 3 drops of NaOH soln
- pH adjustment & sonication seemed to ~~suggest~~ encourage dissolution of pellet. Soln transparent and nearly colourless - hinge of red-purple.

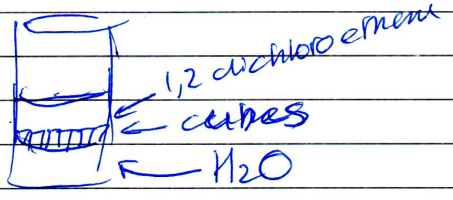
119849

TOPIC: Interface creation and results.

NAME: Michael Walton DATE: 25/1/18

Interface creation:

- Addition of 750 µL of H_2O with dissolved NaCl of various concentrations. to a clean vial. ~~750µL of~~ 250 µL of Au-[] added to vial. then interface created by adding 1,2-dichloroethane.



Ratio of ~~Et~~ 1,2-dichloroethane, cubes & H_2O taken from paper.[1]

Results:

[1. Little if any colour was observed at the interface even after mixing. (Shaken according to ref-1.)] *

2. No apparent difference between NaCl concentrations.

Discussion:

1. Perhaps there was too little salt or too few Au-[] to observe the colour or to bring particles to interface.

1. L. Velleman, D. Sikdar, V.A. Turek, A.R. Kucernak, S.J. Roser, A.A. Kornyshev, J.B. Edel. Nanoscale 2016, 8, ~~14~~ 19229 - 19241.

119850

TOPIC: Discussion & NaCl Soln.

NAME: Michael Walton. DATE: 25/1/18

II. Observed colour on eppendorf looks as if some particles may have crashed (colour still present after soln. was removed to second clean eppendorf.)

III. There was still some colour in the second eppendorf ⇒ some particles crashed & others remained in soln. (?).

NaCl. Soln.:

Want: 20, 40, 60, 80 & 100 mM

in 5mL ⇒ $n = [\ ](5e^{-3})$

$\therefore m = M[\ ](5e^{-3})$

Ideal masses:

0.00585g 0.01168g 0.0175g 0.02336g
0.0292g ⇒ I, II, III, IV, V respectively.

Actual masses	Actual Vol.	Amount used
0.0076g (I)	6.50mL H_2O	750 µL
0.0153g (II)	6.55mL H_2O	750 µL
0.0183g (III)	5.22mL H_2O	750 µL
0.0292g (IV)	6.25mL H_2O	750 µL
0.0299g (V)	5.12mL H_2O	750 µL

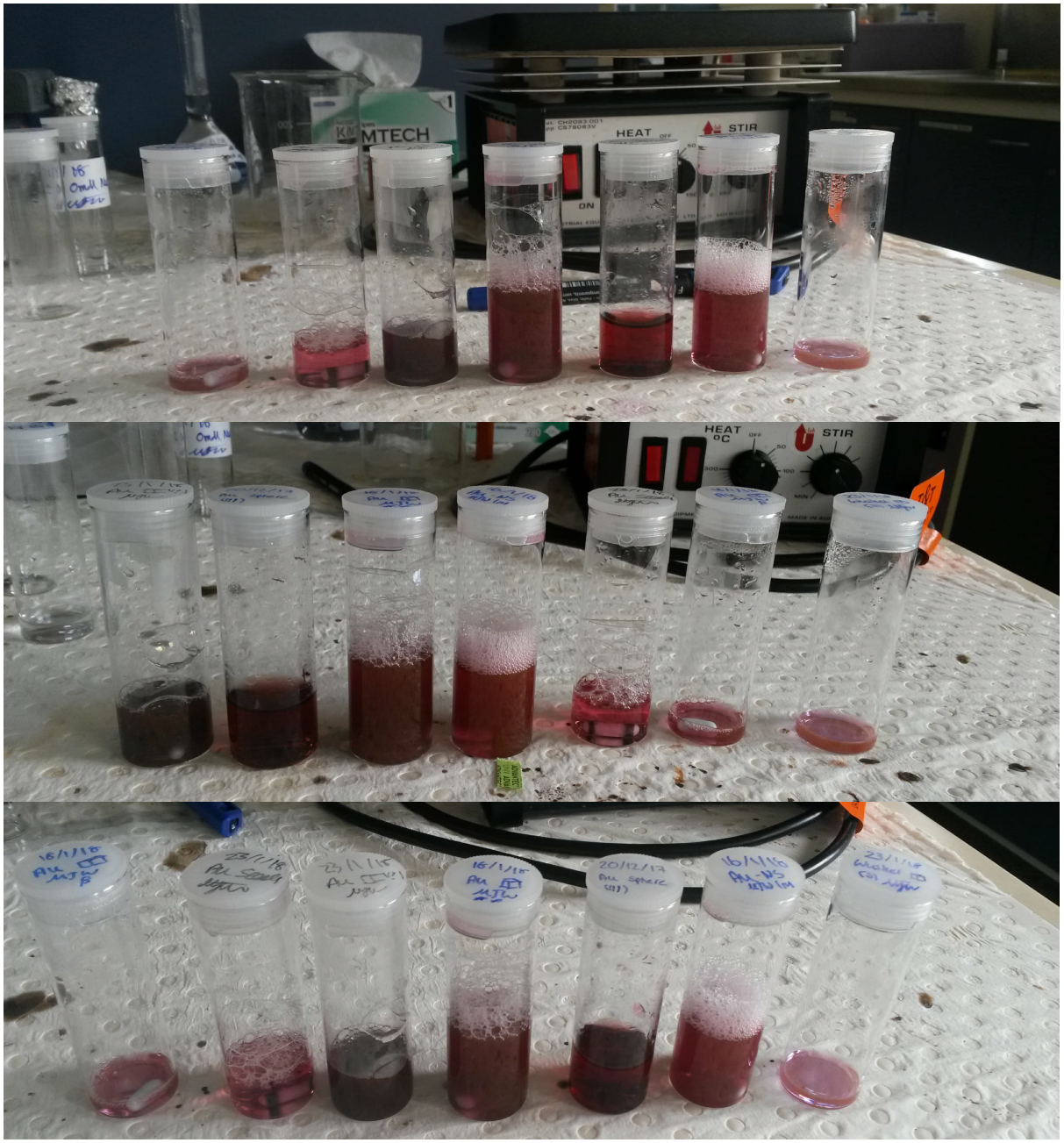


Figure 2: Photograph of gold nano-particle solutions on laboratory work bench. (Top; Middle; Bottom; Left). Taken by M. Walton, summer 2017-18.

Note: The top photo is Fig. 1 but reproduced. It is unclear if each of these photos are different solutions.