1DOC7

Copies of Researchers' Laboratory Notebooks

11/1/09 – 1/7/10

1DOC7

Part A. Preston Brown

9/9/09

Washed carbon nanotube gels
Washed ferrite gels
Vented gels in extractor Rula - CuZn
Shawn - CNT Al

Tom made Iron gels in water

Attended Dr Shi Seminar

9/10/09

Washed carbon nanotube and ferrite gels
XRD analysis on Ferrites

#1,5 - ZnFe	Start 5	Div 2/3°
#2,6 - CoFe	Stop 80	DvHL 10mm
#3,7 - CuFe	Sampling W 0.02	Sct 2/3°
#4,8 - NiFe	Speed 2	Rec S 0.30
		[illegible]

[illegible] still not working will check tomorrow

	Zn	Co	Cu	Ni	
[illegible]	✓	✓	✓	○	In addition
DMSO	✓	✓	✓	✓	[illegible]
Water	✓	✓	✓	✓	[illegible]
Ethanol	✓	✓	✓	○	

1. Gas must be ON before instrument.
2. Regulator at 10 psi (dont mess with regulator valve)
   to switch off.
3. Boot the software
4. Power on. side
5. TPRW — signal — the flowing ① Green.
   — Tells the status.
6. 30 mins of warm up time.
   TCD — 150 mAmps (milliamps).
   TCD — sensitivity — step up or down attenuation
7. Choose function — Reduction
   — Ti at 400, flow for 1/2 hr.
8. CO Pulse titration. (2) Left to right.
   Reduction (3) 1, 2, 3
   AUX — No function.
9. Reduction at higher attenuation

10. Pulse titration — Co on surface
— Very sensitive
— Low attenuation.

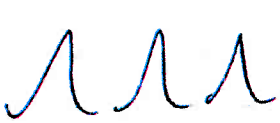
11. Attenuation goes to all the way up.

12. Do zero adjust.

13. Solenoid — automates titration.
(not on ours)

14. 50, 100, 200 µl loops inject.

15. 2 ways — loop
— Gas syringe.

16. 5-10 pulses  3 peaks same height
— titration done
— do average.

17. Wrench for solenoid loop
— Be careful — can get stripped easily.

3-14

18. Take sample — treat with helium (clean)
— reduce
— titrate with CO

19. Every under
— Data acquisition.
— Manual analysis (always for titration)

* titration is NOT MANUAL
TPR is automated.

2 stages — Automatic sample prepation.
— Titrate manually.

Sample prep (Build a macro)

(1) change Gas — Helium
(2) Hit OK
(3) Ramp temp by rate (20 degree per min target 120)
(5 min target)
(4)

(Steps given at manual)

⇒ Manual Analysis
— Titration.
Go to your attenuation &
Use coarse & fine to zero it
hit again.

4-14

- Zero adjust for ∞
- Coarse & fine — Sample or Fine attenuation
- Do the adjust just before sample analysis.

Out gas station of left side (Opp to power switch).
Hear click for release.

Flow rate between 80 & 90 on bubble meter.

Calibration gas done not flow through. — put sample inbetween
- adjust 1-2 bubble every 2 seconds.

Open screens — get thermocouple out (DON'T BEND IT)
- load sample

0.98 ~~23~~ 7

Data acquisition — ChemBET NO
Win X — Instrument set up — Pat

5-14

- CO gas to titration. not the parts.

Data Acq — ChemBET
— Analysis program

Susan or Steven } for esoft question Quantachrome.

My dad has this factorial design

WOW

How can silver just make the structure?

$AgCl_{(s)}$ in H_2O.

$AgCl$

Ag^+ catalyst in presence of HCl?

7-14

Microporous mesopor Mater.

#1 Sako

Silver ions and nanoparticles

TiO_2 - p25?

$O_2 \rightarrow O_2^-$

keep electrons & holes apart.

Ag + ZnO

Role of Silver Ni^{2+}/Ni^0 Pd^{2+}/Pd^0

Microporous zeolite

TiO_2 clean quantum wires

Si Matrix

Ti

Slurry Reactor ETS-10 is visible

multipole plasmon resonance @ 350 nm

500 W Xenon Lamp

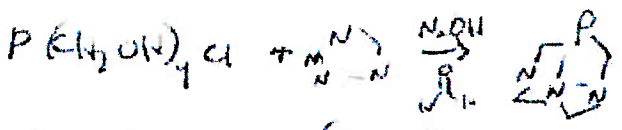
pH ~ 5.2

photo assisted reaction

Brian Frost Nevada Reno

Heterogeneous catalysis is preferred to homogeneous cancer catalysis

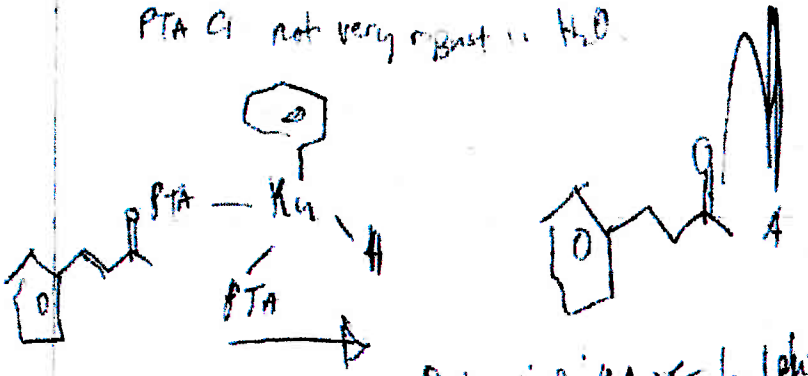
water soluble phosphines.
ligand TPPTS
PTA



Ru · PTA arene (Dyson)
anti cancer agents

PTA can bind by either N or P depending on metal.

PTA Cl not very robust in H_2O



Potential MOF building Block.

9-14

Oct 14 2009

Trial synthesis of Bifetraazole i

- 0.484 g KCN + 1.64 g K_2CO_3 add 3 [illegible]
- 0.51 g NaN_3 in 1.3 mL H_2O
- Add 17 mg $CuSO_4$

42 mg HAc.

After removal from ice and stirring yellow/orange liquid darkened to green/black rxn flask was warm possible need to slow rxn rate by temp control

pH check during synthesis

10-14

11/11

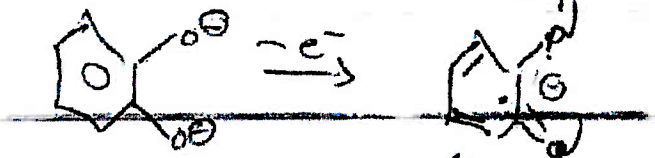
Alan Heida UC Irvine

Metal Catalysis – Metal Redox

d^0 square planar

use d^0 to reduce the ambiguity of where the e^- are being donated by only 1 ligand.

Catechol Ligand



Benzosemiquinone

Stable.

~~Nucleophilic~~ metathesis

12/4

Have made Complexes of

$Co(NO_3)_2$ and hydrazine and

$Ni(NO_3)_2$ and hydrazine

The nickel compound NHN is known and reported but the cobalt compound on the other hand is not reported.
Other metals (Zn, Y, Fe) that we have tried appear to give hydroxide ppt upon addition of hydrazine

12/8/09

Observed formation of two complexes when working with

Co perchlorates:

$Co(NO_3)_2 + NaOH \rightarrow Co(OH)_2$ ppt

$\downarrow HClO_4$

$Co(ClO_4)_2$

red mother liquor yields red crystals after sitting 1-2 days

green powder forms quickly

12-14

Co(II) compounds turn very well

$Co(NO_3)_2$ + hydrazine

8.7 g

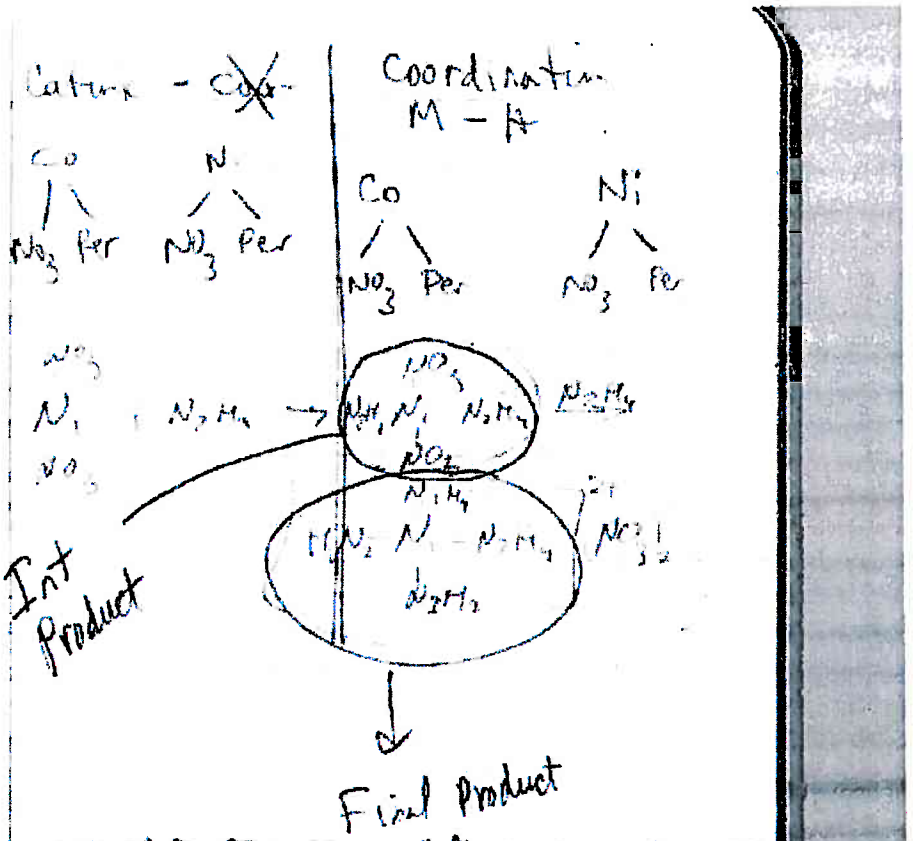
↓

Co hydrazinate nitrate
peach orange color ppt.
exotherm

$Ni(NO_3)_2$ + hydrazine

10 g

purple ppt forms immediately
also an exothermic rxn.



1. Crystal F + Elemental F, I + EC 1 I
   + F
2. (Crystal F) + EC 9 F

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Part B.

Bitetrazole synthesis

KCN 0,5 g dissolved in 10 ml of water, NaN_3 in 10 ml added with stirring, + several drops of acetic acid and 20mg of $CuSO_4$ (cat). Temp 5-8C. Heats on its own, don't let to heat up to much. Leave at 45C for 40 minutes, then heat to 90 for 4 hours.

Heated vigorously developing dark green colour , colour change: green at 45 to blue after 3-4 hours at 90C. Crystals of mixed K Na salt filtered from the mother liquor. NH_3 added , NH_4 salt less soluble than Na filtered after that. Yield ~60%.

Doesn't burn, doesn't work on drop-hammer..

Repeated twice with 0,5g of KCN. Regardless of what we do, heats when we take it out of the ice bath. It looks like it influences the result.

2-aminopyridine

0,5 g of pyridine + 1,1 excess of NaNH2, in 10 ml of toluene, reflux 4 hours. Black resin. Add water to precipitate product, no result.

Repeated 2 times with bigger excess 2.5 of amide and Fe(NO3)3 as catalyst. No product.

Amide looks bad, doesn't really react with water.

Reordered amide

NHN Nickel hydrazine nitrate

0.5g $Ni(NO_3)_2$ reacting with hydrazine (excess, until precipitation stops)~ 2-3ml of hydrazine. Immediately forms pink suspension, filtered, yield is high, but hard to tell, stochiometry is unknown. Insoluble in water, ethanol, acetone, DMFA.

Burns nicely.

Drophammer full height full weight works 50%

CHN

Same as NHN, suspension forms slowly, orange colour, yield less than NHN, takes some time and stirring for precipitation to occur, but still insoluble in all solvents.

Burns.

Works on drophammer with the same weight an height as NHN.

CuHN

Cu nitrate reacts with hydrazine immediately. Managed to get CuHN in alcohol ice bath at around -5C upon treatment with cold diluted hydrazine. Dark blue precipitate, burns nicely but worse than NHN. Colour of flame greenish yellow. Maybe hydrazine perchlorate will be good for fireworks..

Decomposes when dry overnight. Useless.

Other metal hydrazine nitrates

Fe, Zn, Al give precipitates looking very like hydroxides. Do not burn, do not dissolve.

NHP $(NiClO_4)_2$ hydrazine

0,5 g $Ni(NO_3)2$ treated with NaOH to get NiOH. Add $HClO_4$ to dissolve precipitate, controlling pH. Hydrazine added, solution changes colour to green, then blue(precipitate forms), then purple, precipitate dissolves.

Both Blue and purple dried up, both burn. Blue one explodes on the match when compressed.

CHP $(CoClO_4)_2$ hydrazine

0,5 g $Co(NO_3)_2$ treated as Ni, to get perchlorate, then hydrazine added. Colour changes to green, precipitates, does not burn. Too little, no more than 10% If add more hydrazine colour slowly changes to pink, no precipitate. Let it dry up. Product burns and sparkles on the match

CHP works on Drophammer, minimal weight, maximum height (50%)

Flame test good. NHP doesn't burn on flam test.

Tried TGA for CHP, broke the wire, 5 mg too much for it. Apparently exploded at 180C

~25mg spark in mortar!!! Apparently should be ground wet. No problems with grinding wet.(water added, couple drops) But some of it dissolves.

Dissolves in water and hydrazine. trying to get crystals.

Got crystals from water and hydrazine mixture. Took one week, then precipitated overnight.

$Cu(ClO_4)_2$ Hydrazine

Cannot get even at -8C. Reacts immediately with any concentration of Cu and hydrazine.

Flame tests

4 hydrazinates tested on for flame propagation. NHN and CHP ~2m/s, CHN 0.5m/s. NHP sparks or explodes but doesn't burn steadily. Results and video: folder Alex\Flame test.

Amination of bypyridine

0.5g of bypyridine with 2.2 excess of $NaNH_2$ in 10 ml of toluene 12 hours. New amide reacts with water. Distilled toluene before reaction. After reaction no bypyridine left (TLC) but bad mixture of many products. Nothing got as crystals.

Metal bitetrazolates

0.2g of Cu, Ni, Co nitrates treated with bitetrazole + couple of drops of HNO_3, got Blue, dark blue and orange precipitates. All spark on the match, do not really burn, do not work on drophammer with full weight and height. Maybe need more weight.

Diazominotetrazole

Plastic vial!! 0.2 g of 5-aminotetrazole dissolved in 5 ml of 10% acetic acid, treated with equimolar $NaNO_2$ in 5 ml of water. $NaNO_2$ added at ice bath temp, slowly with stirring. Left for 20 mins, then 0.2 more g of 5-aminotetrazole added slowly at 0C.

Cu salt

Olive green precipitate formed after addition of Cu nitrate. Burns.

Will be interesting to try with Ni and Co.

NHP exact procedure

of $Ni(NO_3)_2$ reacted with of NaOH. Precipitate centrifuged, water decanted, then precipitate washed with distilled water, again centrifuged. Repeated 3 times. $Ni(OH)_2$ reacted with HClO4 , pH checked all the time to avoid adding too much acid (and potential formation of hydrazine perchlorate afterwards). Solution transferred to plastic weighing container and there treated with hydrazine. Upon adding blue colour develops and precipitation occurs. At this stage divided into 2 container, one container treated with excess of hydrazine until complete dissolution of precipitate. Colour changes to purple. Both container left overnight in the hood to evaporate solvent without heating. Second Day. In both containers have some solvent left. In the first container, which had less hydrazine initially added hydrazine until precipitates becomes purple and no change occurs upon further addition. So, at this point first container contained **NHP** + **hydrazine** + left from previous day **water** and looked as wet as it might seem.