



SUMITOMO CHEMICAL INDIA LTD.

Research and Development Internship at Mumbai R&D

From 3rd June 2024 to 5th July 2024

Submitted By-

Name – Ram Ratan Aggarwal

2nd Year B.Tech ChE

IIT Kanpur

Signature

Mr. Ram Aggarwal



Signature

Dr. Vandana Mhatre

SUMITOMO CHEMICAL INDIA LTD.
13 & 14, ARADHANA INDUSTRIAL DEVELOPMENT
CORPORATION, NEAR VIRWANI INDUSTRIAL ESTATE,
GOREGAON (EAST), MUMBAI-400 063.
PHONE-42522200/FAX-28713037.

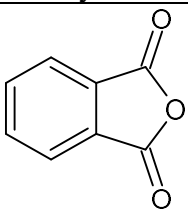
Table of Contents

RAW MATERIALS USED	4
Phthalic Anhydride	4
Hydroxylamine Salts.....	5
FINAL PRODUCT PROPERTIES.....	6
<i>N</i> -hydroxyphthalimide	6
Chemical Reaction Involved.....	7
EXPERIMENTS	7
Experiment 1 – RA-NHPI-1 (05/06/2024).....	7
Experiment 2 – RA-NHPI-2 (06/06/2024).....	10
Purification by Column Chromatography of the sample (12/06/2024).....	11
Experiment 3 – RA-NHPI-3 (07/06/2024).....	12
Recrystallisation of NHPI collected from RA-NHPI-1 and RA-NHPI-3	14
Experiment 4 – RA-NHPI-4 (10/06/2024).....	15
Experiment 5 – RA-NHPI-5 (11/06/2024).....	17
Experiment 6 – RA-NHPI-6 (12/06/2024).....	20
Purification of PA by Recrystallisation:	21
Experiment 8 – RA-NHPI-8 (17/06/2024).....	23
Block wise Procedure	24
Experiment 9 – RA-NHPI-9 (17/06/2024).....	25
Experiment 10 – RA-NHPI-10 (18/06/2024).....	26
Experiment 11 – RA-NHPI-11 (20/06/2024).....	28
ANALYSIS.....	30
UV Spectroscopy	30
UV Spectroscopy of PA-RC	30
UV Spectroscopy of RA-NHPI-11	31
High Performance Liquid Chromatography	32
HPLC Conditions used (Method of Analysis)	32
HPLC of PA-RC	33
HPLC of RA-NHPI-CC-2.....	34
HPLC of RA-NHPI-1+3-RC.....	35
HPLC of RA-NHPI-05-Crude	36

HPLC of RA-NHPI-08-Crude	37
HPLC of RA-NHPI-11-Crude	38
HPLC Results.....	38
SUMMARY	39
OBSERVATIONS & CONCLUSIONS:	40
ABBREVIATIONS:	40
BHAVNAGAR PLANT VISIT(24/06/24 to 28/06/24):.....	41
REFERENCES	44

RAW MATERIALS USED

Phthalic Anhydride

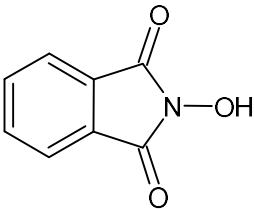
IUPAC NAME	2-Benzofuran-1,3-dione
MOLECULAR FORMULA	C ₈ H ₄ O ₃
COMMON NAME¹	Phthalic anhydride
STRUCTURE	<div></div> <p>2-benzofuran-1,3-dione</p>
MELTING POINT	130.8°C
MOLECULAR WEIGHT	148.12 g/mol
APPEARANCE	White lustrous needles like crystals
BOILING POINT	295°C
DENSITY	1.53 g/m ³
MANUFACTURING/PRODUCTION	<p>Phthalic Anhydride is prepared from naphthalene by oxidation with a mixture of HgSO₄ and CuSO₄ in presence of H₂SO₄; by passing naphthalene and oxygen over a suitable catalyst at 400-500°C.</p> <p>Also manufactured by Dehydration of Phthalic Acid</p>
PRECAUTIONS	Wear protective gloves/eye protection/face protection as on overexposure causes irritation of eyes, skin and upper respiratory system. Can leads to conjunctivitis, nasal ulcer bleeding, bronchitis, bronchial asthma, dermatitis.
USES	Phthalates (esters of phthalic acid), benzoic acid, synthetic indigo, artificial resins (glyptal).
CAS NO.	85-44-9
MANUFACTURER NAME	Anmol Chemical Molecules Pvt. ltd.
MP (EXPERIMENTAL)	131.5°C – 133.0°C
SOLUBILITY IN WATER	0.62 gram/100 grams (20-25°C)
METHOD OF ANALYSIS	GC & HPLC

Hydroxylamine Salts

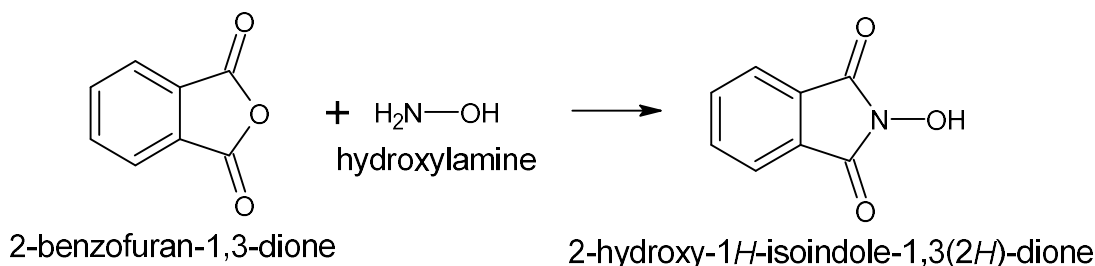
IUPAC NAME	Hydroxylamine hydrochloride	Hydroxylammonium sulfate
MOLECULAR FORMULA	ClH ₄ NO	[NH ₃ OH] ₂ SO ₄
COMMON NAME	Hydroxylammonium chloride (HAC)	Hydroxylammonium sulfate (HAS)
STRUCTURE	[NH ₃ OH] ⁺ Cl ⁻	([NH ₃ OH] ⁺) ₂ [SO ₄] ²⁻
MELTING POINT	155 to 157 °C	120 °C Decomposition
MOLECULAR WEIGHT	69.49 g/mol	164.14 g/mol
APPEARANCE	Colourless solid	Colourless solid
DENSITY	1.67 g/cm ³	1.88 g/cm ³
MANUFACTURING/PRODUCTION	Preparation done in Hydrochloride (HCl) known as Oxammonium hydrochloride (H ₃ NO.HCl). It is monoclinic columnar crystals; slowly decays when there is moisture.	Preparation done in Sulphuric Acid (H ₂ SO ₄) known as Oxammonium Sulfate (H ₃ NO) ₂ -H ₂ SO ₄ . It is crystal in nature
PRECAUTIONSⁱⁱ	Obtain special instructions before use and read all safety precautions thoroughly. Use the required personal protective equipment (PPE) and wash your face, hands, and any exposed skin after handling.	Obtain special instructions before use and read all safety precautions thoroughly. Use the required personal protective equipment (PPE) and wash your face, hands, and any exposed skin after handling.
USES	As a reducing agent in photography, in synthetic and analytical chemistry, to purify ketones and aldehydes, as antioxidant for fatty acids and soaps, as a dehairing agent for hides.	As a reducing agent in photography, in synthetic and analytical chemistry, to purify ketones and aldehydes, as antioxidant for fatty acids and soaps, as a dehairing agent for hides.
CAS	5470-11-1	10039-54-0
MANUFACTURER NAME	MERCK	SD Fine chemicals Ltd.
SOLUBILITY IN WATERⁱⁱⁱ	84g/100 mL (20°C)	58.7 g/100 mL (20 °C)
METHOD OF ANALYSIS^{iv}	IR Spectroscopy	IR Spectroscopy

FINAL PRODUCT PROPERTIES

N-hydroxyphthalimide

IUPAC NAME	2-Hydroxy-1 <i>H</i> -isoindole-1,3(2 <i>H</i>)-dione
MOLECULAR FORMULA	C ₆ H ₄ (CO) ₂ NOH
COMMON NAME^v	<i>N</i> -Hydroxyphthalimide
STRUCTURE	
MELTING POINT	233°C with decomposition
MOLECULAR WEIGHT	163.132 g/mol
APPEARANCE	White to pale yellow crystalline solid
BOILING POINT	370°C
DENSITY	1.64 g/m ³
POLYMORPH DIFFERENCE	<i>N</i> -Hydroxyphthalimide exists in two polymorphs, colorless and yellow, In the colorless white form, the NOH group is rotated about 1.19° from the plane of the molecule, while in the yellow form it is much closer to planarity (0.06° rotation).
PRECAUTIONS	Wear appropriate protective eyeglasses or chemical safety goggles and Wear appropriate protective gloves and clothing to prevent skin exposure
USES	Serves as intermediate for one agrochemical molecule, A variety of functional groups can be oxidized with the aminoxyl radical (phthalimide- <i>N</i> -oxyl), used for generating active esters from <i>N</i> -hydroxyphthalimide
CAS NO.	524-38-9
MANUFACTURER NAME	Spectrochemicals India ltd.
SOLUBILITY IN WATER	5.05 gram/100 mL (20-25°C)
METHOD OF ANALYSIS	HPLC

Chemical Reaction Involved



EXPERIMENTS

AIM – To synthesize N-Hydroxy Phthalimide(NHPI) from Phthalic Anhydride(PA) and Hydroxylamine Sulfate (HAS) or Hydroxylamine Chlorate (HAC).

APPARATUS USED- 100 mL 4-necked RBF, Magnetic needle for stirring, Thermocouple, Separation funnel, Condenser

Experiment 1^{vi} – RA-NHPI-1 (05/06/2024)

	PA	HAS	NaOH	NHPI
Mol. Wt.	148.12	164.14	40	163.13
Purity (%)	99.0	97.0	97.0	-
Molar ratio	2	1	2	2
Moles	0.01	0.005	0.01	-
Moles Taken	0.01	0.0055	0.012	-
Amount required	1.49	0.93	0.494	-
Amount taken	1.5	0.93	0.51	-

PA + HAS + Water was added in one go in required amount and then NaOH was added and heated till 100°C and cooked at 100°C for 1 hour and after cooking the precipitate was filtered through Buchner funnel under vacuum to get NHPI crude material. The 2nd crop was isolated from the filtrate by extracting the filtrate with ethyl acetate using separating and was evaporated using Rotary Evaporator.

SEQUENTIAL PROCEDURE-

Time (in Hours)	IN Temp. (° C)	OUT Temp. (° C)	Remarks
12:45	31	-	PA + HAS + Water(5mL) was added in RB under continuous stirring. Suspension formation took place inside the RB, hence

			we added 5mL more water and started heating to 50°C.
12:50	52	59	NaOH solution prepared in 4mL of water and the added to the RBF. On addition there is color change is observed from colorless to light yellow. And heating slowly raised to 100°C.
12:55	58	85	Almost clear with slight small solids seen. Color very light Yellow was observed.
13:00	71	94	Temperature was raised to 100°C. Very minute white colored precipitate was observed in the solution. The color of solution was light yellow
13:05	80	106	Precipitate formation took place inside the RBF. White Color Precipitate was seen.
13:12	96	121	pH of the Reaction Mixture was checked using pH paper and it is observed to be around 5. Mild Reflux was seen
13:18	99	125	Cooking Started by maintaining Temperature at 100° C
13:55	101	128	White Colored Precipitate observed in the RBF having yellow colored solution.
14:20	100	122	TLC Sample of the Reaction Mixture after 1 hour. Liquid was removed by dropper then water (2 mL) added pH is around 5 is observed. Ethyl Acetate (2 mL) added extraction done. Water removed by dropper & EtOAc dried with anhydrous Na ₂ SO ₄ & spot on TLC. Solvent system (Hexane = 2 mL ; EtOAc = 3 mL; Methanol = 1 drop)
14:50	70	30	RD is taken out of the oil bath and kept for cooling. Precipitate formation takes place.
15:10	19	RT	Precipitate separated out through vacuum pumping and washed with the filtrate that is collected.
15:26	RT	RT	Collected Precipitate is kept in the oven for drying
15:54	RT	RT	The Product is removed from the oven and weight of the product is measured.
16:00	RT	RT	Melting point of the Product is measured using capillary.

Extraction Procedure-

Separation of residue from filtrate is done with the help of separating funnel and rotary evaporator through the following steps:-

1. The filtrate first collected in a test tube and then mixed with ethyl acetate (5 mL) and transferred into separating funnel, we choose ethyl acetate because NHPI is more soluble in ethyl acetate and ethyl acetate is immiscible in water so created a separate layer and we separate out that layer one of water and another one of ethyl acetate and collect in different beakers.
2. pH of the water separated out was checked and it was found slightly acidic* (so we have to wash it again till the solution became somewhat neutral) so we give it a wash i.e adding of ethyl acetate (3 mL) and performed the same procedure as we did in first 0step.
3. After separation the collected 3mL ethyl acetate is mixed with initially collected 5mL solution and collected in separating funnel and give it a wash of water (around 5mL) Now we separate out ethyl acetate containing NHPI and water in separate beaker.
4. If some amount of water came with ethyl acetate layer during separation so we add on sodium sulfate as it absorbed all the moisture present in the solution and we filter out it later and collected solution was taken for the distillation in rotary evaporator to separate out the remaining product from the filtrate.

ANALYSIS-

Amount of NHPI filtered out – 0.73 grams

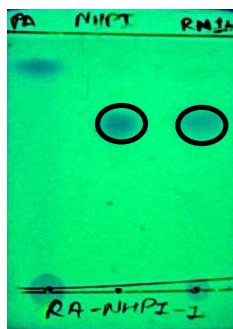
Theoretical Amount of NHPI that had to be formed – 1.6313 grams

Yield - 44.75 %

Melting Point of the sample – 223°C

Appearance – Yellow

TLC^{vii}- We had taken solvent system as Hexane = 2 mL ; EtOAc = 3 mL; Methanol = 1 drop.
Marking first point as PA, 2nd as NHPI and 3rd as Reaction Mixture after cooking



Experiment 2^{viii} – RA-NHPI-2 (06/06/2024)

	PA	HAS	NaOH	NHPI
Mol. Wt.	148.12	164.14	40	163.13
Purity	99%	97%	97%	-
Molar ratio	2	1	2	2
Moles	0.01	0.005	0.01	-
Moles Taken	0.01	0.0055	0.012	-
Amount required	1.49	0.93	0.494	-
Amount Taken	1.5048	0.9405	0.4607	-

First we prepared NaOH solution in water then poured in the 4-necked 100 mL RBF, after that we added HAS in the specified quantity and wait till we got cleared solution and then PA was added to the solution, then heated to 100°C and kept for cooking for an hour. After completion of cooking, the reaction mixture was cooled and then filtered out, to obtain wet NHPI, which was dried in oven till constant weight. Melting point was taken.

First, we measured the quantity of the chemicals. Then we prepared a solution of NaOH in 3 mL and poured it into the RBF. After that, we added 2 mL more water to clean the beaker. HAS was gradually added to the RB flask, and the mixture was stirred well and heated until a clear solution was obtained. After getting a clear solution, we slowly added phthalic anhydride to the reagents with continuous stirring and raised the temperature to 100°C. At 45-50°C, we observed a thick viscous white mixture inside the RBF, but after raising the temperature to 60-65°C, it became smooth and turned into a pale yellow suspension, and it was easy to stir. The temperature was raised to 100°C, and cooking continued for 1 hour. We gradually cooled the RBF to 10°C and obtained a yellow precipitate inside the RBF. We then filtered it using a Buchner funnel and vacuum pump, and the filtrate collected was used to wash the RBF 1-2 more times. The pH of the filtrate was analyzed and found to be acidic, around 4-5. We also washed the solid with water, collected all the filtrate into a test tube, and separated the residue in a petri dish, which was kept for drying in an oven at 60°C for 30-40 minutes. The collected sample was filled in a capillary tube and analyzed for melting point.

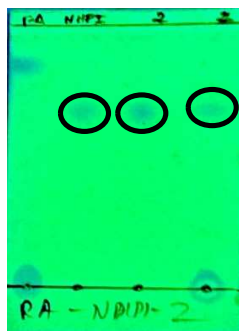
Observed MP – 229.8°C

Weight of the collected sample – 1.21 gm

Calculated Yield – 74.12 %

Appearance - Lime Yellow crystalline solid

TLC - We had taken solvent system as Hexane = 2 mL ; EtOAc = 3 mL; Methanol = 1 drop. Marking first point as PA, 2nd as NHPI, 3rd as Reaction Mixture after cooking and 4th as RM of RA-NHPI-1.



Purification by Column Chromatography of the sample (12/06/2024)

We purified this sample (around 1g) and removed the colored and other impurities using column chromatography. To prepare the column, we first added silica gel 60-120 mesh (around 48g) in hexanes and poured it into the column with continuous tapping so the silica gel settled uniformly. Then we made a slurry of the sample by the following method. We added 1g of our sample and dissolved it in ethyl acetate (19 mL), warmed it to 75°C, and obtained a light yellow colored clear solution. Then we added silica gel (6g) and evaporated the solvent on a rotary evaporator at 65°C under vacuum (300-400 mbar) to get a mango-colored slurry, which was loaded with hexane onto the column. Then the following solvent system was used along with the timestamp.

Time	Ethyl Acetate (in mL)	Hexane (in mL)	Methanol (in mL)
2:15 PM	45	5	-
2:34 PM	40	10	-
2:46 PM	35	15	-
2:54 PM	30	20	-
3:01 PM	25	25	-
3:31 PM	20	30	-
3:36 PM	15	35	-
3:46 PM	10	40	-
4:09 PM	0	100	-
4:40 PM	0	49	1

We collected the sample in different test tubes and checked it using TLC and isolated the pure NHPI using rotary evaporator @65°C under vacuum between 200-300 mbar. After evaporating the solvent we got yellow crystals.

Weight of NHPI sample Taken: 1.0000 grams

Weight of NHPI crystals we got: 0.9353 grams

TLC- - We had taken solvent system as Hexane = 2 mL ; EtOAc = 3 mL; Methanol = 1 drop.



Experiment 3^{ix} – RA-NHPI-3 (07/06/2024)

	PA	HAS	NaOH	NHPI
Mol. Wt.	148.12	164.14	40	163.13
Purity	99%	97%	97%	-
Molar ratio	2	1	2	2
Moles	0.01	0.005	0.01	-
Moles Taken	0.01	0.0055	0.012	-
Amount required	1.49	0.93	0.494	-
Amount Taken	1.5038	0.9449	0.4490	-

We also prepared another solution of NaOH containing 0.5247 grams of sodium hydroxide in water, weighing 5.27 grams for maintaining the pH. First, we added the NaOH solution to the RB flask and continued heating. Then, HAS was added, and PA was added when HAS became properly soluble in NaOH. We checked the pH of the solution and found it very acidic, around 2-3. We then added the NaOH solution we prepared separately dropwise until the pH reached around 5-6, which is slightly acidic. The mixture was cooked for 1 hour and then filtered under vacuum.

Procedure-

1. We took the mentioned quantity for the reaction, prepared a solution of NaOH (0.449 grams) in 3 mL water, and poured it into the flask. We washed the beaker with an additional 2 mL of water, using a total of 5 mL of water.
2. We added HAS (0.9449 grams) to the RB flask and started heating until we obtained a clear solution. Then we added phthalic anhydride to the solution, slowly raising the temperature to 100°C with continuous stirring. The resulting solution was pale yellow, and its pH was found to be very acidic, around 2-3. We then added the NaOH solution dropwise until the pH became slightly acidic, around 5-6.
3. We then cooked the mixture/slurry at 100°C for 1 hour. We observed the formation of a very thick pale yellow slurry, which became less thick as the temperature rose. To check the completion of the reaction, we performed TLC on the reaction mixture.
4. For TLC, we prepared a solution of the reaction mixture in ethyl acetate under slow heating. We also prepared solutions of the starting material (phthalic anhydride) and the final standard (NHPI) in ethyl acetate, marking their spots on the TLC strip using a capillary tube. We used a mixture of hexane, ethyl acetate, and methanol in a ratio of 40%, 60%, and 1 drop, respectively.
5. After confirming completion of the reaction from TLC, we allowed the reaction mixture to cool until the temperature reached 14°C. The formed slurry was filtered out using a vacuum pump. To recover the remaining solid in the RBF, we washed the RBF first with the mother liquor (filtrate) and then with 5mL of cold water.
6. Afterward, we checked the pH of the solution and found it to be acidic. We washed it with normal tap water until pH 6, then placed the filter wet cake in the oven to dry for half an hour.
7. After drying, we weighed the sample and found it to be around 0.7347 grams (yielding only 45.17%).

To check the melting point, we filled the sample into an MP capillary tube and obtained a melting point of 223.2°C. To trace the remaining material or product, we analyzed the mother liquor and separated out the product and other materials using a separation funnel and rotary evaporator, as done previously.

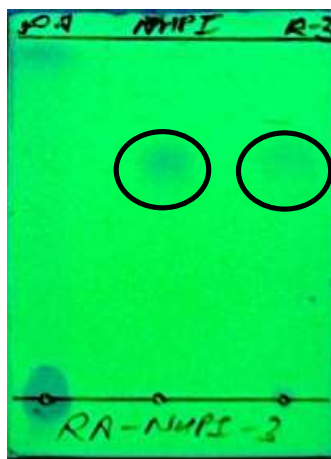
Observed MP – 223.2°C

Weight of the collected sample – 0.7347 gm

Calculated Yield – 45.17 %

Appearance – Yellow colored

TLC- - We had taken solvent system as Hexane = 2 mL ; EtOAc = 3 mL; Methanol = 1 drop. Marking first point as PA, 2nd as NHPI, 3rd as Reaction Mixture after cooking



Recrystallisation of NHPI collected from RA-NHPI-1 and RA-NHPI-3

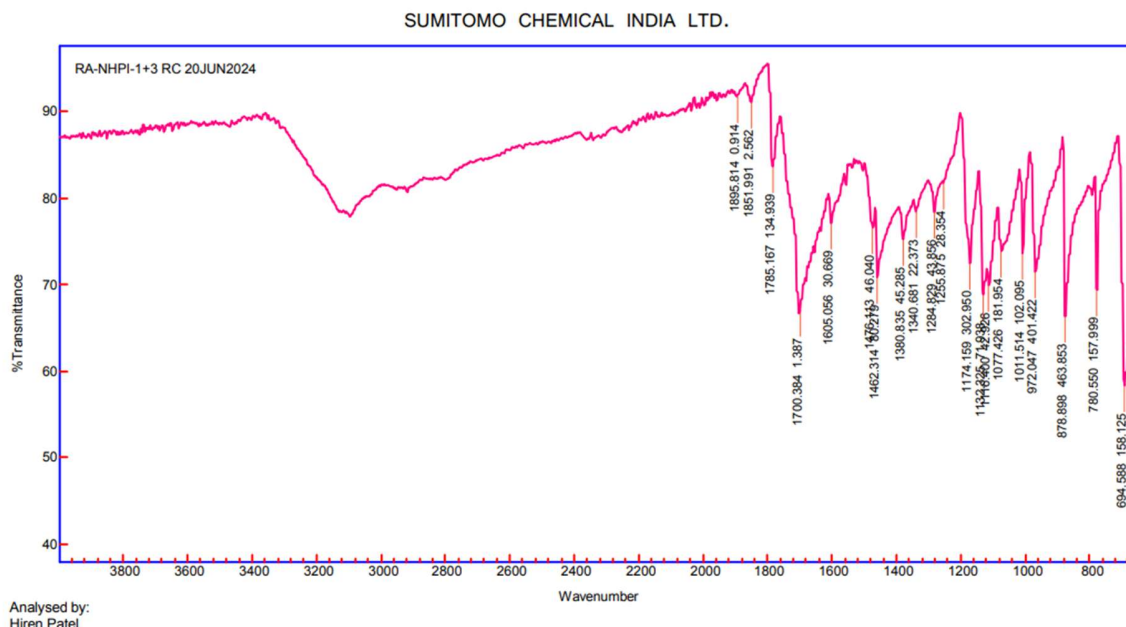
We took the samples collected from RA-NHPI-1 and RA-NHPI-3 and mixed them together for recrystallization, as their melting points did not differ significantly, indicating they likely contained similar types or compositions of impurities.

For recrystallization, we prepared a saturated solution of the NHPI (1+3) in methanol and kept it at room temperature. After some time, we observed crystals starting to form, so we left it overnight to obtain finer crystals, which were filtered and dried.

Collected crystal weighs 0.7964 grams.

Observed MELTING POINT = 132.2°C

IR SPECTROSCOPY OF COLLECTED CRYSTALS



RA-NHPI-1+3 RC 20JUN2024

Experiment 4^x – RA-NHPI-4 (10/06/2024)

	PA	HAC	Na ₂ CO ₃	NHPI
Mol. Wt.	148.12	69.49	105.98	163.13
Purity	99%	98%	98%	-
Molar ratio	2	2	1	2
Moles	0.01	0.005	0.01	-
Moles Taken	0.0153	0.0196	0.0206	-
Amount required	2.266	1.362	2.183	-
Amount Taken	2.2887	1.3941	2.2283	-

First, we took the raw materials in the desired quantities for the reaction, using water as a solvent, Na₂CO₃ is taken in excess so that HAC reacts completely and PA got enough reagent for reaction. We prepared HAC solution in water and heated it to 55°C, then added Na₂CO₃. After complete dissolution, we added phthalic anhydride and cooked the mixture at 60°C for one hour before filtering it out.

Time	In Temp. (°C)	Out Temp. (°C)	Remark
10:11 AM	27	29	We added 5mL water with HAC and kept it for heating till 55°C.
10:19 AM	52	72	Na ₂ CO ₃ weight is calculated
10:28 AM	55	62	Na ₂ CO ₃ added and there is slow drop in temperature was observed, maybe reaction was endothermic.
10:50 AM	63	70	PA was added to the solution, along with 1 more mL of water because the solution became turbid. After the addition of PA, a white-colored solution formed with some effervescence of CO ₂ . The pH of the solution was found to be basic.
10:53 AM	60	68	Thick slurry formation took place of orange color. We added 4 mL of water and mix it properly. After that a yellow colored solution formation with some precipitate formation.
10:55 AM	60	67	Cooking started. pH found between 6-7

After 1 hour of cooking, the RB was kept for cooling and chilled to 7°C. We prepared a 1:1 aqueous HCl solution and added it dropwise until the dark yellow/orange color disappeared. After continuous stirring, we filtered out the NHPI under vacuum and placed the wet cake in the oven for drying. Finally, we checked the melting point of the NHPI.

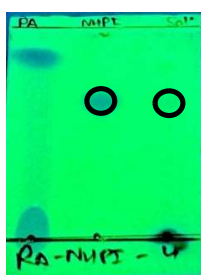
Observed MP – 223°C

Weight of the collected sample – 2.4958 gm

Calculated Yield – 5.40 %

Appearance – White

TLC- We used a solvent system consisting of 2 mL of hexane, 3 mL of ethyl acetate (EtOAc), and 1 drop of methanol. The first spot was marked as PA, the second as NHPI, and the third as the reaction mixture after cooking.



Experiment 5^{xi} – RA-NHPI-5 (11/06/2024)

	PA	HAS	NaOH	NHPI
Mol. Wt.	148.12	164.14	40	163.13
Purity (%)	99.0	97.0	97.0	-
Molar ratio	2	1	2	2
Moles	0.0297	0.015	0.03298	-
Moles Taken	0.0298	0.0154	0.03372	-
Amount required	4.4436	2.538	1.36	-
Amount taken	4.4651	2.6053	1.3905	-

First, we prepared a NaOH solution in water and warmed it. After that, we added HAS in the specified quantity and waited until we obtained a clear solution. Then, we added PA in equal intervals and amounts with a spoon, heating the solution to 65°C and cooking it for an hour. After completing the cooking process, we filtered out the NHPI and dried it to measure its weight and melting point.

Firstly, we measured the amount of raw materials needed as mentioned above. We prepared a NaOH solution containing 1.3905 grams of NaOH in 10mL of water and poured this solution into the RB flask, warming it. Then, we added 2.6053 grams of HAS, stirred it well, and added another 5mL of water. We heated the sample to 65°C and then added phthalic anhydride in the following manner.

Time	In Temp.(°C)	Out Temp. (°C)	Remark
10:36 AM	65	75	1 spoon of PA added to the solution under continuous stirring and heating.
10:41 AM	66	75	While heating and stirring continuously, 1 spoon of PA was introduced to the solution.
10:47 AM	67	77	We added 1 spoon of PA to the solution, maintaining constant stirring and heating.
10:52 AM	66	73	1 spoon of PA added to the solution under continuous stirring and heating.
10:57 AM	66	77	1 spoon of PA added to the solution under continuous stirring and heating.
11:02 AM	65	73	1 spoon of PA added to the solution under continuous stirring and heating.
11:15 AM	65	74	Cooking started along with formation of thick slurry.

After completion of cooking we turn off heating and kept RBF in room temperature so that it attain some ambient temperature then cooled it using chilled water resulting in precipitating out NHPI from the RM. So we filtered out the reaction mixture using filter paper under vacuum and collected wet cake kept in oven for drying. We obtained crystalline cotton like white colored NHPI through this process and calculated its melting point. We also did IR spectroscopy of the material that formed.

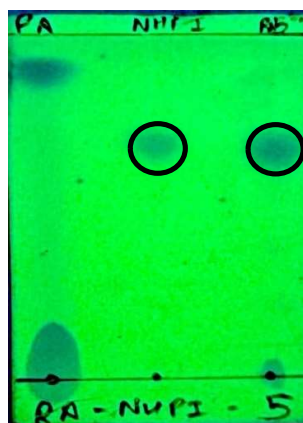
Observed MP – 228.8°C

Weight of the collected sample – 3.4558gm

Calculated Yield – 70.64%

Appearance – White

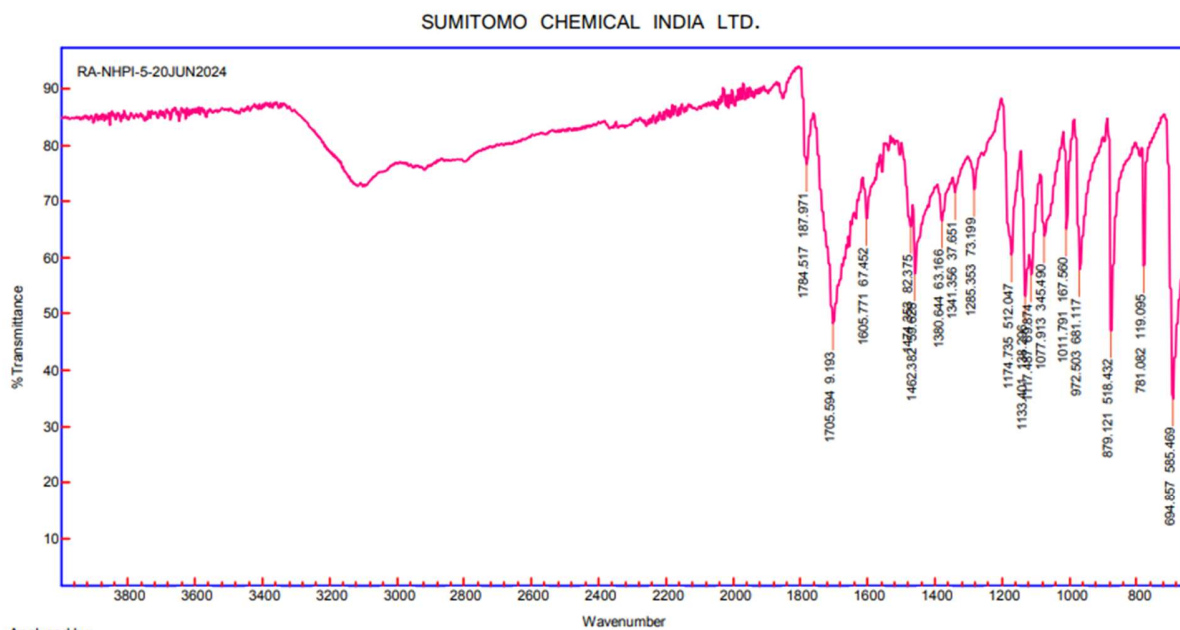
TLC- We used a solvent system consisting of 2 mL of hexane, 3 mL of ethyl acetate (EtOAc), and 1 drop of methanol. The first spot was marked as PA, the second as NHPI, and the third as the reaction mixture after cooking.



During TLC we got to know that PA contains little bit amount of impurity because of the spot as seen at the bottom first spot, maybe it undergoes hydrolysis and formed phthalic acid also in the reaction mixture spot of salt is seen

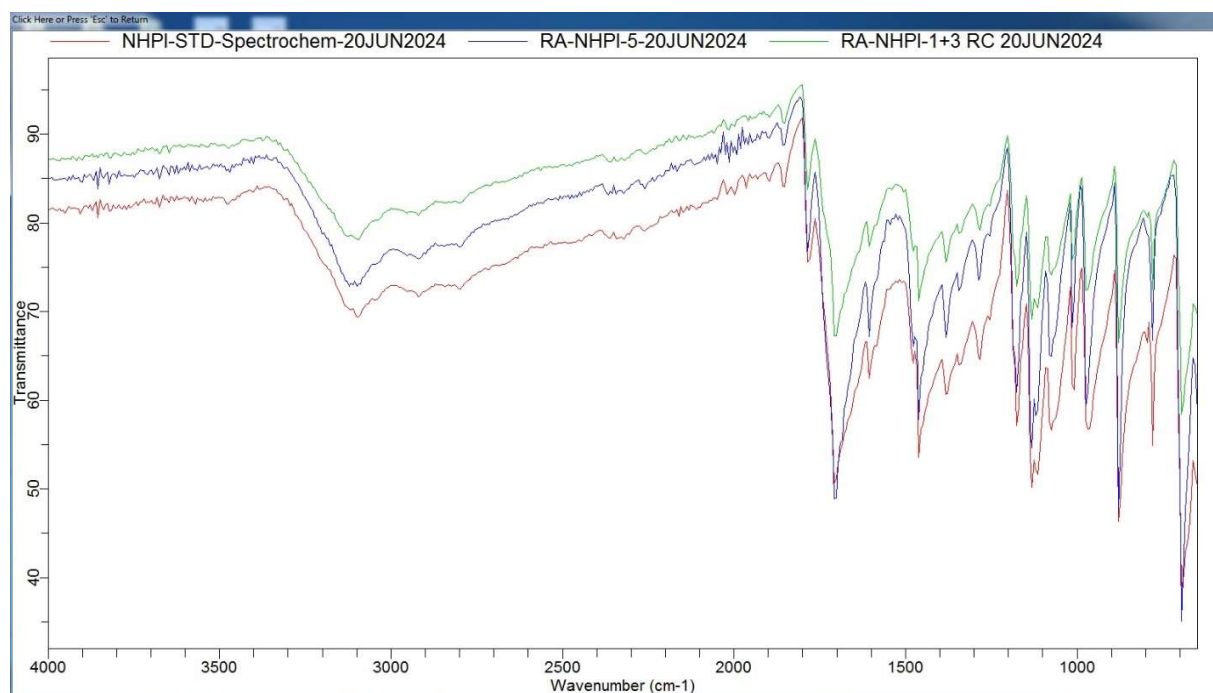
IR spectroscopy of the material

White and Yellow polymorphs form of NHPI shows same kind of IR spectroscopy there is only difference in projection of their structure, In white form the N-OH group is rotated about 1.19° from the plane of the molecule, while in yellow form it is much closer to planarity. (0.06° rotation)



RA-NHPI-5-20JUN2024

We also compared IR graph of RA-NHPI-5 and RA-NHPI-1+3-RC with the fine NHPI present in lab



Through above graphs from IR spectroscopy we can say that synthesized compound matches with fine compound, therefore final compound formed is NHPI.

Experiment 6^{xii} – RA-NHPI-6 (12/06/2024)

	PA	HAS	NaOH	NHPI
Mol. Wt.	148.12	164.14	40	163.13
Purity (%)	99.0	97.0	97.0	-
Molar ratio	2	1	2	2
Moles	0.037	0.020	0.040	-
Moles Taken	0.037	0.022	0.041	-
Amount required	5.57	3.78	1.70	-
Amount taken	5.58	3.78	1.6993	-

NaOH and HAS were taken in excess so that PA would react completely. First, we prepared a NaOH solution in water and warmed it briefly. Then, we added HAS in the specified quantity and waited until we obtained a clear solution. Next, we added PA to the solution, heated it to 100°C, and cooked it for an hour. After cooking, we filtered out the NHPI, dried it, and measured its weight and melting point.

First, we measured the quantity of the chemicals. Then we prepared a NaOH solution in 15mL of water and poured it into the RB flask. After that, we added 5mL more water to clean the beaker. Next, HAS was gradually added to the RB flask, and the mixture was stirred well and heated until a clear solution was obtained. After obtaining a clear solution, we slowly added phthalic anhydride to the reagents with continuous stirring and raised the temperature to 100°C. Upon addition, we observed the formation of a yellow solution. At 45-50°C, a thick, viscous, light yellow mixture formed inside the RBF, but as the temperature increased to 60-65°C, it became smooth and pale yellow, with continuous stirring. The temperature was then raised to 100°C, and the mixture was cooked for 1 hour. We gradually cooled the RBF to 10°C and obtained a yellow precipitate inside the RB. The mixture was filtered using a Buchner funnel and vacuum pump, and the filtrate was used to wash the RB 1-2 more times. The pH of the filtrate was found to be acidic, around 4-5. We also washed it with 35mL of water, and the pH of the collected filtrate was found to be 3-4. The collected residue was separated in a petri dish and kept in an oven at 60°C for 30-40 minutes. The dried sample was then filled into a capillary tube and analyzed for its melting point.

Observed MP –233.3°C

Weight of the collected sample – 4.26gm

Calculated Yield –70.14 %

Appearance – light yellow

TLC- We used a solvent system consisting of 2 mL of hexane, 3 mL of ethyl acetate (EtOAc), and 1 drop of methanol. The first spot was marked as PA, the second as NHPI, and the third as the reaction mixture after cooking.



Purification of PA by Recrystallisation:

We took a sample of phthalic anhydride weighing 10.0316 grams and dissolved it in ethyl acetate to obtain a saturated solution. This solution was kept overnight to allow fine crystals of phthalic anhydride to form.

We obtained very fine, needle-like crystals of phthalic anhydride that are white in color. We also analyzed the sample using GC and found its purity to be around A% = 99.96%.

We added ethyl acetate in the following order:

first 10mL under continuous heating and stirring, then an additional 15mL. Since a large amount of PA remained insoluble, we added another 5mL, followed by 15mL. We observed that a small amount of PA was still insoluble and settling at the bottom, so we added another 5mL twice. In total, we added 55mL of ethyl acetate for recrystallization and collected the 1st and 2nd crops, which weighed as follows.

Weight of 1st crop of RC : 5.281 grams

Weight of 2nd crop of RC : 2.6718 grams

MP of PA : 132.4°C

Experiment 7^{xiii} – RA-NHPI-7 (14/06/2024)

	PA	HAS	NaOH	NHPI
Mol. Wt.	148.12	164.14	40	163.13
Purity (%)	99.9	97.0	97.0	-
Molar ratio	2	1	2	2
Moles	0.01	0.005	0.01	-
Moles Taken	0.01016	0.0055	0.011	-
Amount required	1.4812	0.0056	0.0113	-
Amount taken	1.5052	0.9411	0.4653	-

In this reaction, we used recrystallized PA to ensure the purity of the PA used. First, we prepared a NaOH solution in water, then added HAS. After obtaining a clear solution, we added PA and cooked it for 1 hour at 100°C. We then filtered out the precipitate formed using a vacuum pump and Buchner funnel.

Procedure for the reaction:-

1. First, we carried out calculations to determine the required amounts of HAS and NaOH for the given amount of PA, ensuring HAS and NaOH were in excess so that all the phthalic anhydride would react effectively.
2. We prepared a NaOH solution in an RB flask containing 0.4653 grams of NaOH and 3mL of water. We then washed the NaOH beaker with 2mL of water and poured it into the RBF, warming the solution.
3. We added HAS to the NaOH solution and heated it in an oil bath under continuous stirring until we obtained a clear solution.
4. At around 10:31 AM, when the internal temperature was 50°C and the oil bath temperature was 82°C, we added the recrystallized PA to the RB and slowly raised the temperature. At around 65°C, we observed the formation of a pale yellow solution, and at around 70°C, a very thick slurry material formed, preventing the needle from rotating. However, after reaching 80-85°C, it became smoother, and stirring continued.
5. We raised the temperature until the flask reached 100°C and at 11:05 AM, we cooked the reaction mixture at 100°C for 1 hour.
6. After cooking we cooled down the RM to 8°C, filtered out the product formed using Buchner After cooking, we cooled down the reaction mixture to 8°C and filtered out the product using a Buchner funnel and vacuum pump.
7. The collected mother liquor was used to wash the RBF, followed by cold water (around 6°C) to wash the RBF.
8. We washed the cake with tap water until pH 6.
9. The wet cake was kept in an oven for drying, and after drying, we carried out the melting point and weight of the collected sample.

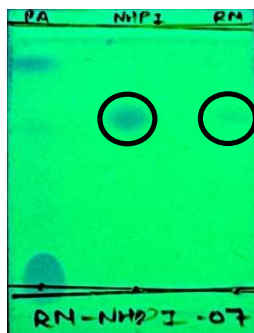
Observed MP –229.8°C

Weight of the collected sample – 0.9028gm

Calculated Yield –55.3%

Appearance – Yellow

TLC- We used a solvent system consisting of 2 mL of hexane, 3 mL of ethyl acetate (EtOAc), and 1 drop of methanol. The first spot was marked as PA, the second as NHPI, and the third as the reaction mixture after cooking.



Experiment 8^{xiv} – RA-NHPI-8 (17/06/2024)

	PA	HAS	NaOH	NHPI
Mol. Wt.	148.12	164.14	40	163.13
Purity (%)	99.9	97.0	97.0	-
Molar ratio	2	1	2	2
Moles	0.01	0.005	0.01	-
Moles Taken	0.020	0.011	0.022	-
Amount required	2.9624	1.881	0.9214	-
Amount taken	2.9688	1.889	0.9220	-

In this reaction, we used recrystallized PA to ensure the PA's purity. First, we prepared a NaOH solution in water, then added HAS. After obtaining a clear solution, we added PA and cooked it for 1 hour at 100°C. We then filtered out the precipitate using a vacuum pump and Buchner funnel. This reaction was similar to the previous one, but we increased the quantity of raw materials and used 10mL of water. The procedure and experimental steps were the same as mentioned above.

By separating NHPI in ethyl acetate from the mother liquor and then distilling that solution under vacuum using a rotary evaporator, we found its weight to be 0.22 grams. We also performed distillation of the separated water containing salt to study the mass balance and determine where the remaining yield was going. The weight of the salt formed was found to be around 1.4664 grams.

Observed MP –228.7°C

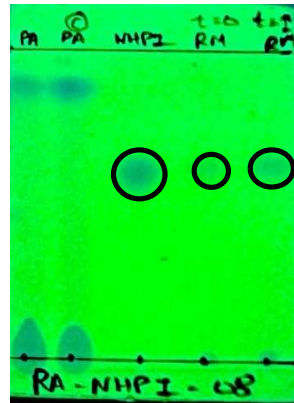
Weight of the collected sample – 2.4796 gm

Calculated Yield –76 %

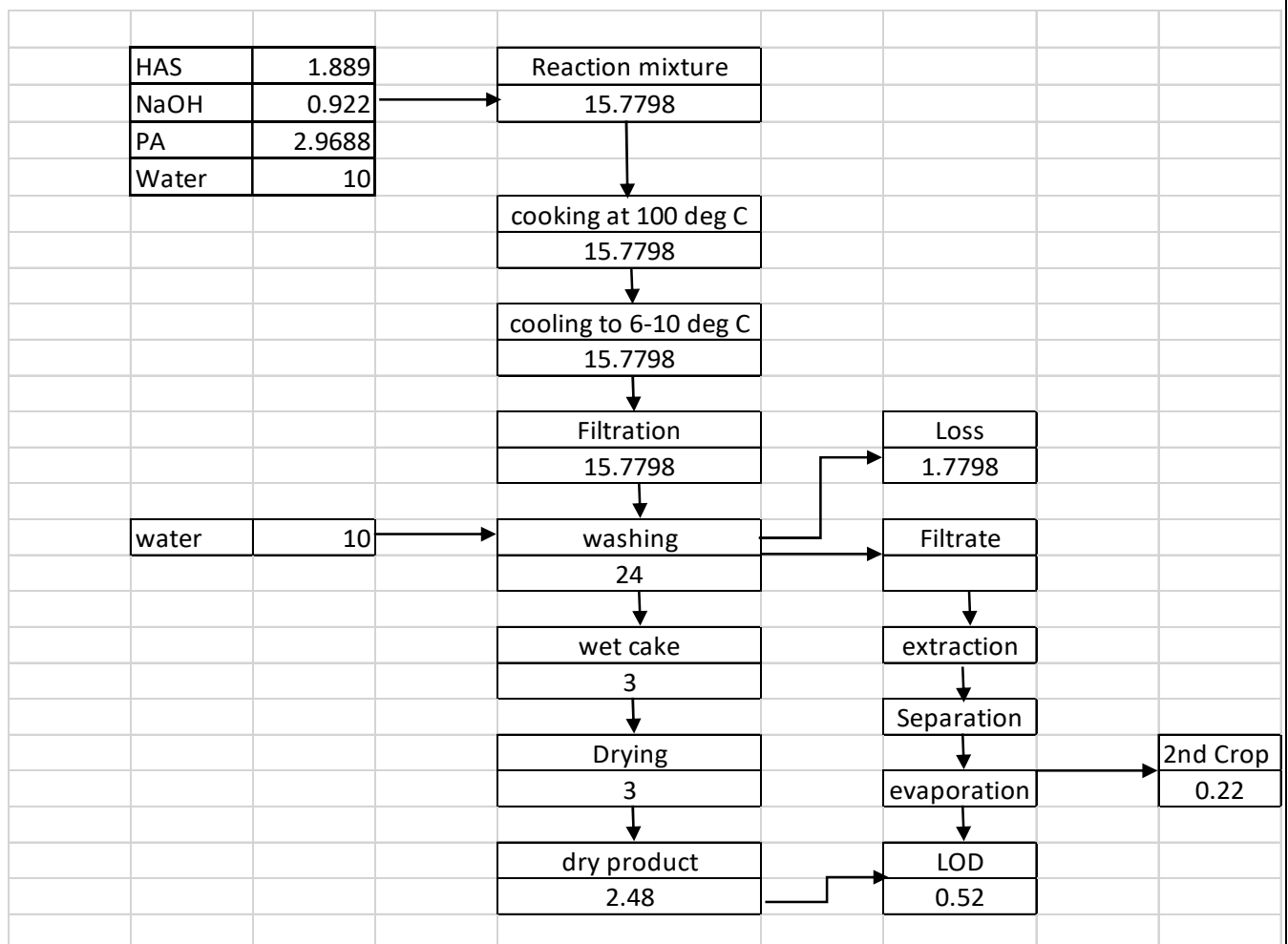
Appearance – Yellow crystalline

TLC- We used a solvent system consisting of 2 mL of hexane, 3 mL of ethyl acetate (EtOAc), and 1 drop of methanol. The first spot was marked as PA, the second as PA-Crystalize, the third

as NHPI, the fourth spot is of the reaction mixture before cooking and the fifth spot is of RM after cooking.



Block wise Procedure



Experiment 9^{xv} – RA-NHPI-9 (17/06/2024)

	PA	HAC	Na ₂ CO ₃	NHPI
Mol. Wt.	148.12	164.14	40	163.13
Purity (%)	99.9	97.0	97.0	-
Molar ratio	2	1	2	2
Moles	0.01	0.005	0.01	-
Moles Taken	0.020	0.011	0.022	-
Amount required	2.77	1.3	2.2	-
Amount taken	2.779	1.3176	2.22	-

The solvent used is toluene, with a volume of 5mL. It's known that inorganic salts are insoluble in toluene, yet we proceeded with this reaction.

Experimental Procedure:-

1. Firstly, we added 5mL of toluene to the RB flask and heated it until it reached 55°C. Once the temperature reached 55°C, we added HAC, and the solution became almost clear after some time.
2. After achieving a clear solution of toluene and HAC, we added sodium carbonate to the solution. However, sodium carbonate is insoluble in toluene, so we did not observe any reaction taking place inside the RB flask.
3. So we added water to the RB flask using a dropper to initiate the reaction.
4. Just after adding drops of water, we observed the effervescence of CO₂, indicating that the reaction had started and sodium carbonate was reacting with HAC.
5. Then we added PA to the RB flask and raised the temperature to 100°C. During heating, we observed the formation of a red-colored slurry.
6. We cooked the reaction mixture (RM) for 1 hour, then filtered it using a Buchner funnel and vacuum pump.
7. The filtered solid was orange in color, and after keeping the filtrate for some time, a thick red-colored filtrate was collected, with red-colored solids settling down.
8. We separated the water and toluene mixture using a separating funnel because toluene and water formed an immiscible mixture. The red part was soluble in water but not in toluene.
9. After separating the layers of toluene and water, we collected them in separate beakers. The water layer contained a red, sticky substance. We evaporated the water using a rotary evaporator to get reddish substance using a vacuum pump. The samples were subsequently dried in an oven.
10. After completely drying the NHPI, we calculated its weight and determined its melting point (MP) using a capillary tube in an oil bath.

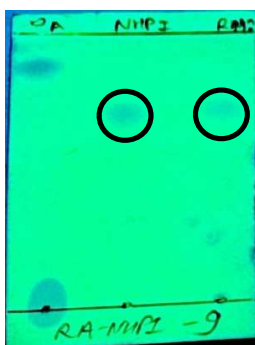
Observed MP – 215°C

Weight of the collected sample – 1.2869gm

Calculated Yield – 39.44 %

Appearance – orange

TLC- We used a solvent system consisting of 2 mL of hexane, 3 mL of ethyl acetate (EtOAc), and 1 drop of methanol. The first spot was marked as PA, the second as NHPI, and the third as the reaction mixture after cooking.



Experiment 10^{xvi} – RA-NHPI-10 (18/06/2024)

	PA	HAC	NaOH	NHPI
Mol. Wt.	148.12	69.49	40	163.13
Purity (%)	99.9	98.0	97.0	-
Molar ratio	1	1	1	2
Moles	0.01	0.01	0.01	-
Moles Taken	0.01	0.0102	0.0102	-
Amount required	1.4812	0.7233	0.42	-
Amount taken	1.4836	0.7282	0.428	-

Theoretical weight = 1.63131 grams

Procedure :-

1. First, we measured the required amount of raw material and took 5 mL of water for the reaction. We then added NaOH solution prepared in that water to the reaction bottle (RB).
2. We heated the mixture to 35°C and added HAC, then waited until we achieved a clear solution. Simultaneously, we increased the temperature while stirring continuously.
3. After obtaining a clear solution at around 55°C, we added PA (collected from RC) to the RB.

4. Just after adding PA, within a few minutes, PA started to react with the reactant, forming a yellow-colored solution.
5. At 71°C, we observed the formation of a very thick, highly viscous slurry in the RBF. We continued to increase the temperature until the internal temperature reached 100°C, while the temperature of the oil bath was around 126°C.
6. After reaching 100°C, the reaction mixture was kept cooking for 1 hour.
7. After 1 hour, the RBF was removed from the oil bath to allow the temperature inside the RBF to slowly cool down to ambient temperature. Then, we placed the RBF in a cold water bath to lower the temperature of the reaction mixture to 6°C.
8. After cooling the reaction mixture, we filtered it using a Büchner funnel and vacuum pump, collecting the mother liquor in a test tube. Some amount of NHPI remained stuck inside the RBF, so we washed it with 5 mL of cold water to remove it, collecting this as additional mother liquor.
9. The pH of the solution was found to be very acidic, so we washed it with cold water in 6 X 5 mL. After washing, we checked the pH of the filtrate and found it to be slightly acidic, almost neutral.
10. The collected wet cake was separated out and placed in an oven for drying. After drying, we measured the weight of the dried cake and determined its melting point.

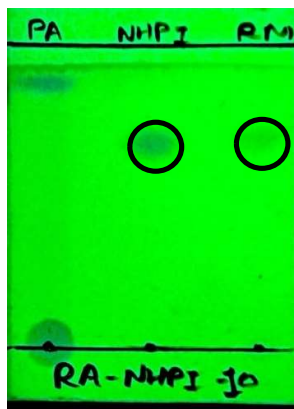
Observed MP -226°C

Weight of the collected sample – 1.14 gm

Calculated Yield – 69.88 %

Appearance – Pale Yellow

TLC - We used a solvent system comprising 2 mL of hexane, 3 mL of ethyl acetate (EtOAc), and 1 drop of methanol. The first spot was labeled as PA, the second as NHPI, and the third as the reaction mixture after cooking.



Experiment 11^{xvixviii} – RA-NHPI-11 (20/06/2024)

	PA	HAC	Na acetate.3H ₂ O	NHPI
Mol. Wt.	148.12	69.49	136.3	163.13
Purity (%)	99.0	98.0	97.0	-
Molar ratio	1	1	1	1
Moles	0.01	0.01	0.01	-
Moles Taken	0.0676	0.0864	0.0864	-
Amount required	10.0	6.0	11.75	-
Amount taken	10.2291	6.0435	11.794	-

Solvent used :- water that is 30mL in volume, Acetic acid that is 80mL in volume

The amount of acetic acid (HAC) and sodium acetate were taken in excess to ensure that PA had enough reagent to react with. The PA used in this reaction was from a bottle already present in the lab, not the PA collected from the reaction mixture (RC).

Procedure:-

1. We prepared a solution of hydroxylamine hydrochloride in 30 mL of water and added it to the RB flask using a funnel. Heating of the reaction mixture began under continuous stirring.
2. Next, we added 11.79 grams of sodium acetate trihydrate to the solution to neutralize the HCl formed from hydroxylamine hydrochloride.
3. After complete dissolution of HAC and sodium acetate, we prepared another solution of PA in 80 mL of acetic acid. PA was found to be sparingly soluble in acetic acid, requiring an excessive amount to dissolve. Due to the impracticality of using large volumes of acetic acid on a laboratory scale, we proceeded with caution.
4. Attempts to add the PA and acetic acid solution dropwise resulted in PA settling at the bottom of the column. Rolling up the heating sleeve along the dropping funnel did not prevent solid settling and blocking the column, so we opted to pour the solution directly.
5. After complete addition, we maintained the temperature at 59 - 60°C and observed a color change in the solution from colorless to yellow.
6. Cooking continued for 5 to 5.5 hours, during which a viscous, pale yellow slurry formed intermittently.
7. After completing the cooking process, we turned off the heating and allowed the RBF to cool to ambient temperature. The reaction mixture was then cooled in chilled water to 8°C.
8. The cooled reaction mixture was filtered using a Büchner funnel and vacuum pump, collecting the mother liquor in a separate flask.
9. The pH of the filtrate was highly acidic, so we washed the wet cake with water in three series: first with 30 mL, then with 25 mL, and finally with another 25 mL of water.

10. The collected wet cake was dried in an oven, and after drying, we measured its weight and melting point. The sample weighed 6 grams.
11. The collected mother liquor was distilled using a rotary evaporator. On distillation, 1.05 grams of solid remained in the RB flask, and 65 mL of solvent was recovered.

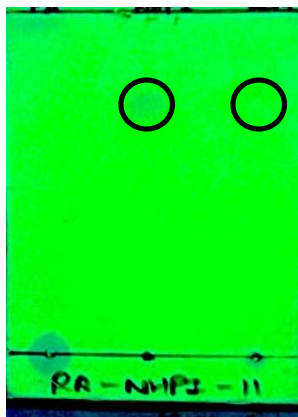
Observed MP – 231°C

Weight of the collected sample – 6 gm

Calculated Yield – 64 %

Appearance – Light Yellow

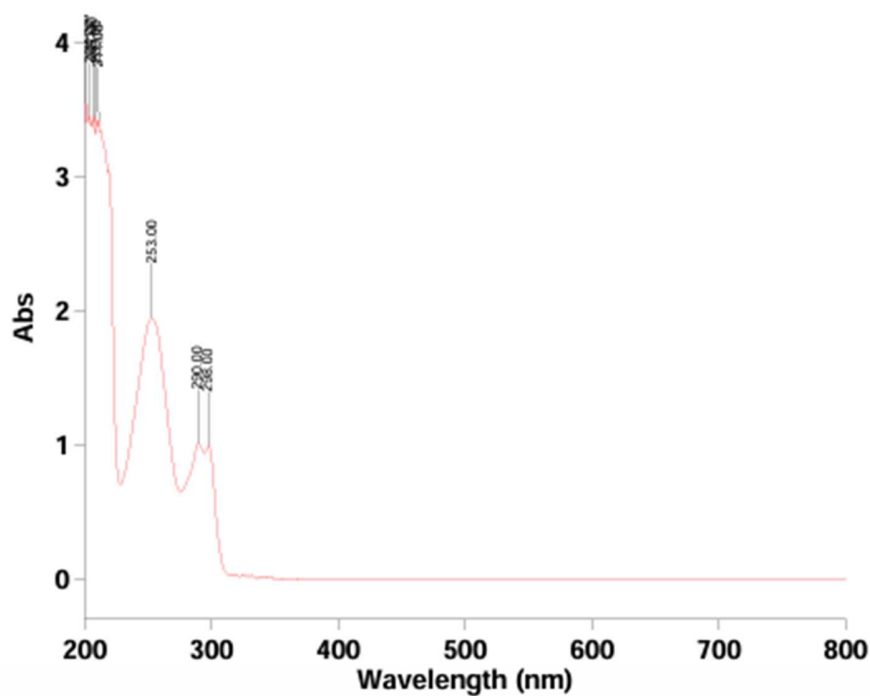
TLC- We used a solvent system comprising 2 mL of hexane, 3 mL of ethyl acetate (EtOAc), and 1 drop of methanol. The first spot was labeled as PA, the second as NHPI, and the third as the reaction mixture after cooking.



ANALYSIS

UV Spectroscopy

UV Spectroscopy of PA-RC



Scan Analysis Report

Report Time : Tue 02 Jul 03:25:40 PM 2024
Method:
Batch: C:\Varian\Cary Winuv\RAM\RA-PA-RC-UV-02Jul2024-trial.DSW
Software version: 3.00(339)
Operator:

Sample Name: RA-PA-RC-UV-02Jul2024

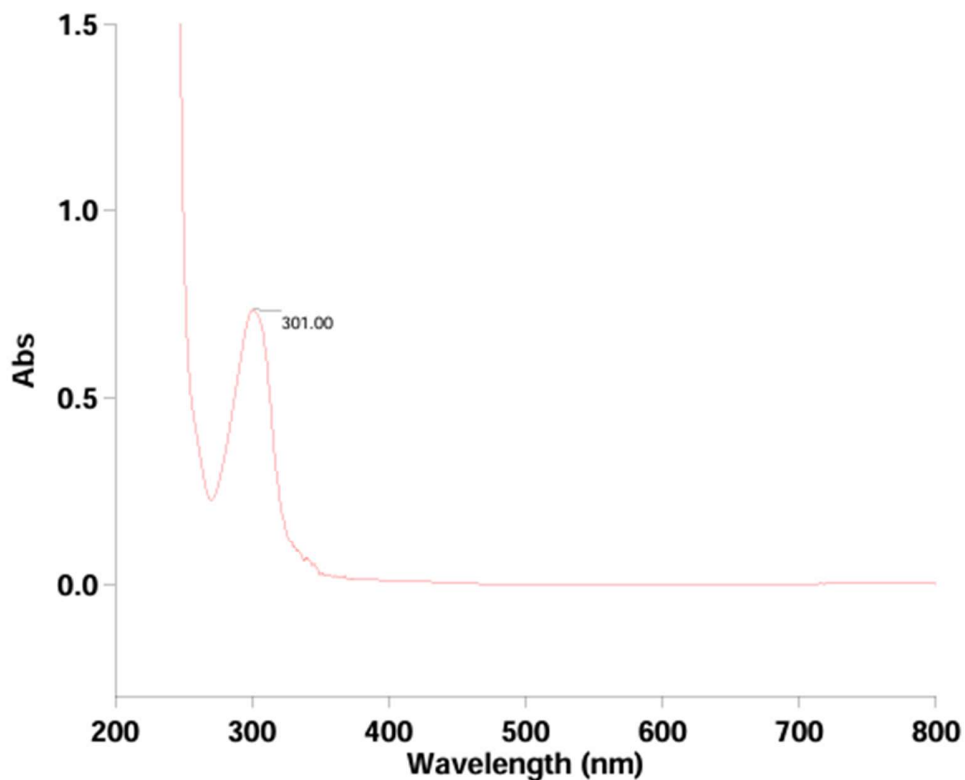
Collection Time 7/2/2024 12:25:35 PM

Peak Table
Peak Style Peaks
Peak Threshold 0.0500
Range 800.00nm to 200.00nm

Wavelength (nm)	Abs
298.00	0.999
290.00	1.015
253.00	1.950
211.00	3.418
209.00	3.441
207.00	3.453
204.00	3.452
201.00	3.551

UV spectroscopy graph for Phthalic Anhydride crystals formed after Recrystallization
 λ max is found out to 253 nm

UV Spectroscopy of RA-NHPI-11



Scan Analysis Report

Report Time : Tue 02 Jul 03:16:59 PM 2024
Method:
Batch: C:\Varian\Cary Winuv\RAM\RA-PA-RC-UV-02Jul2024-trial.DSW
Software version: 3.00(339)
Operator:

Sample Name: RA-NHPI-11-UV-02Jul2024

Collection Time 7/2/2024 11:48:33 AM

Peak Table
Peak Style Peaks
Peak Threshold 0.5000
Range 800.00nm to 200.00nm

Wavelength (nm)	Abs
301.00	0.736

UV spectroscopy graph for N-hydroxy phthalimide crude formed during RA-NHPI-11
 λ max is found out to 301 nm

High Performance Liquid Chromatography

HPLC Conditions used (Method of Analysis)

Method of analysis of NHPI

HPLC System : Agilent 1260 Infinity or equivalent
Column : ZORBAX Eclipse XDB-C18 (4.6mm x 250mm x 5 micron)
or equivalent

Mobile Phase :

Solution A: **Acetonitrile**

Solution D: **Water**

Flow rate : 0.8mL/min

Run time : 30 minute

Post run time : 5 minute

Wavelength : 254nm

Injection volume : 2 μ L

Column temperature : 30°C

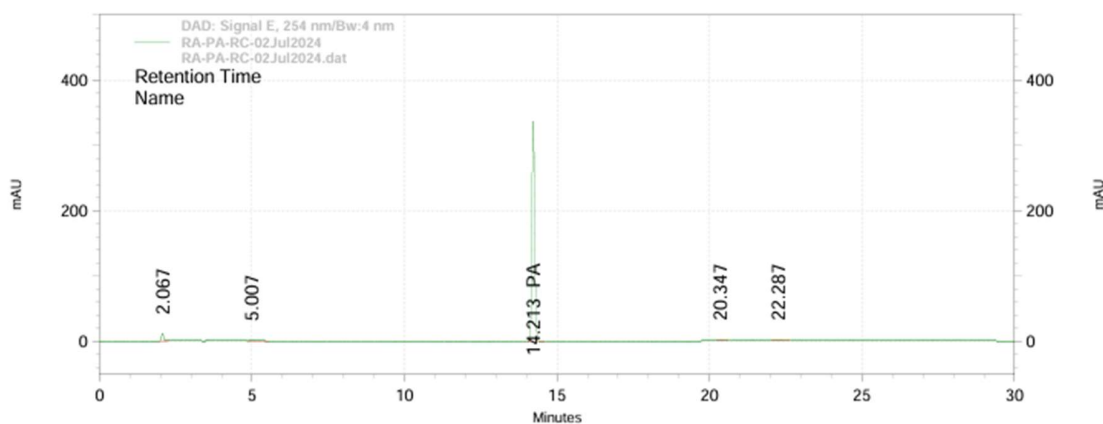
Gradient Condition :

Time [min]	Δ	A [%]	B [%]	C [%]	D [%]	Flow [mL/min]	Max. Pressure Limit [bar]
0.00		20.0	0.0	0.0	80.0	0.800	400.00
5.00		20.0	0.0	0.0	80.0	0.800	400.00
8.00		40.0	0.0	0.0	60.0	0.800	400.00
10.00		50.0	0.0	0.0	50.0	0.800	400.00
15.00		70.0	0.0	0.0	30.0	0.800	400.00
18.00		90.0	0.0	0.0	10.0	0.800	400.00
25.00		90.0	0.0	0.0	10.0	0.800	400.00
30.00		20.0	0.0	0.0	80.0	0.800	400.00

Sample Preparation: Take 25mg of sample in 25mL of volumetric flask and add 15mL of Acetonitrile. Sonicate it to dissolve completely and shake well. Finally, Dilute up to the mark with Acetonitrile. (1000 ppm Concentration)

**MUMBAI
HPLC Report**

Data File: D:\HPLC3\RA-NHPI\Result\NHPI-02Jul2024-ON\RA-PA-RC-02Jul2024.dat
Method: D:\HPLC3\RA-NHPI\Method\RA-NHPI-01.met
Sample I.D: RA-PA-RC-02Jul2024
Acquired: 7/2/2024 6:37:28 PM (GMT +05:30)
Printed: 7/3/2024 10:55:50 AM (GMT +05:30)
Instrument ID: HPLC3
Inj vol: 2ul
Vial No.: 42



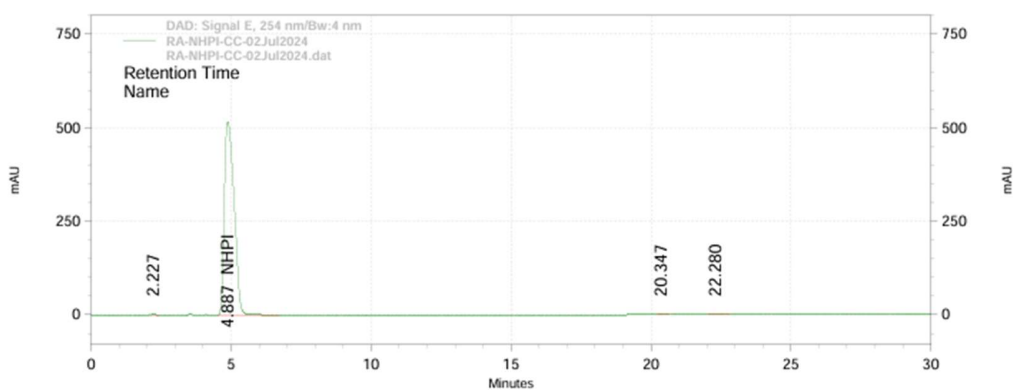
DAD: Signal E, 254
nm/Bw:4 nm Results

Pk #	R T	Name	Area	Area %
1	2.07		139021	3.05
2	5.01		22942	0.50
3	14.21	PA	4346359	95.31
4	20.35		29229	0.64
5	22.29		22574	0.50

Totals			4560125	100.00
--------	--	--	---------	--------

**MUMBAI
HPLC Report**

Data File: D:\HPLC3\RA-NHPI\Result\NHPI-02Jul2024-ON\RA-NHPI-CC-02Jul2024.dat
Method: D:\HPLC3\RA-NHPI\Method\RA-NHPI-01.met
Sample I.D: RA-NHPI-CC-02Jul2024
Acquired: 7/2/2024 7:14:33 PM (GMT +05:30)
Printed: 7/3/2024 10:58:55 AM (GMT +05:30)
Instrument ID: HPLC3
Inj vol: 2ul
Vial No.: 43

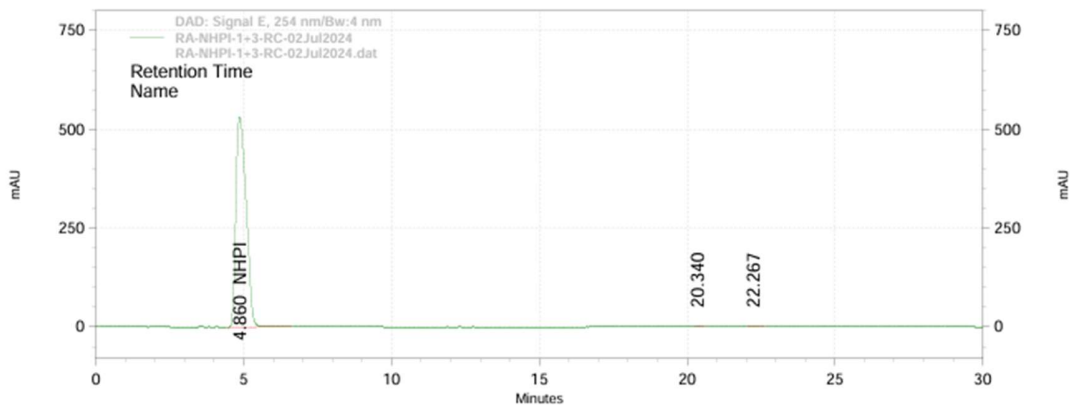


DAD: Signal E, 254
nm/Bw:4 nm Results

NHPI Data File Results				
Pk #	R T	Name	Area	Area %
1	2.23	NHPI	27296	0.10
2	4.89		26197330	99.69
3	20.35		30017	0.11
4	22.28		25005	0.10
Totals			26279648	100.00

MUMBAI
HPLC Report

Data File: D:\HPLC3\RA-NHPI\Result\NHPI-02Jul2024-ON\RA-NHPI-1+3-RC-02Jul2024.dat
Method: D:\HPLC3\RA-NHPI\Method\RA-NHPI-01.met
Sample I.D: RA-NHPI-1+3-RC-02Jul2024
Acquired: 7/2/2024 7:51:36 PM (GMT +05:30)
Printed: 7/3/2024 10:58:18 AM (GMT +05:30)
Instrument ID: HPLC3
Inj vol: 2ul
Vial No.: 44

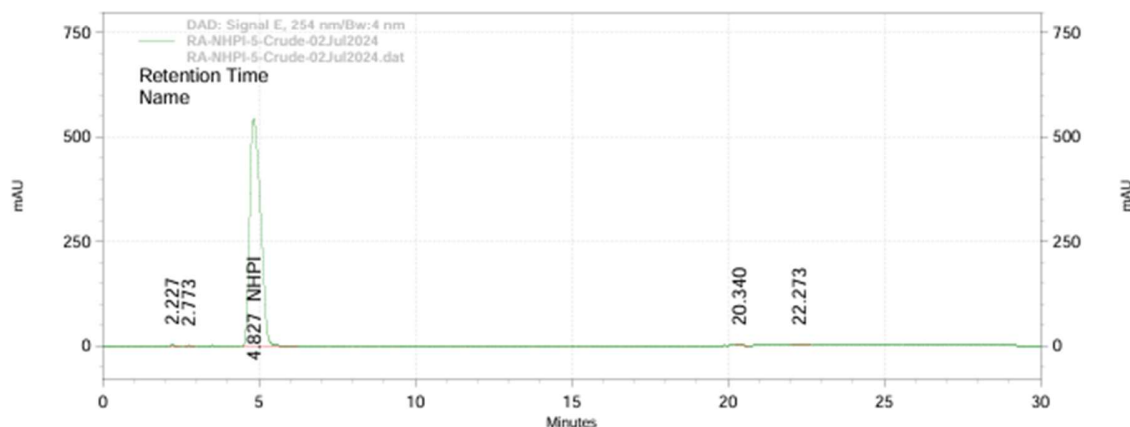


DAD: Signal E, 254
nm/Bw:4 nm Results

Pk #	R T	Name	Area	Area %
1	4.86	NHPI	26978495	99.81
2	20.34		29552	0.11
3	22.27		21676	0.08
Totals			27029723	100.00

**MUMBAI
HPLC Report**

Data File: D:\HPLC3\RA-NHPI\Result\NHPI-02Jul2024-ON\RA-NHPI-5-Crude-02Jul2024.dat
Method: D:\HPLC3\RA-NHPI\Method\RA-NHPI-01.met
Sample I.D: RA-NHPI-5-Crude-02Jul2024
Acquired: 7/2/2024 8:28:41 PM (GMT +05:30)
Printed: 7/3/2024 10:59:50 AM (GMT +05:30)
Instrument ID: HPLC3
Inj vol: 2ul
Vial No.: 45



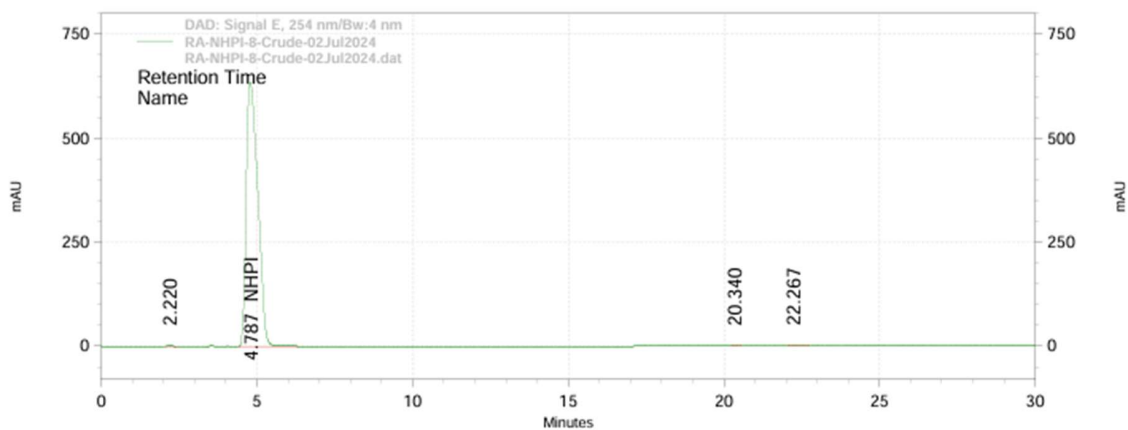
DAD: Signal E, 254
nm/Bw:4 nm Results

Pk #	R T	Name	Area	Area %
1	2.23		44837	0.16
2	2.77		12069	0.04
3	4.83	NHPI	27262338	99.60
4	20.34		29477	0.11
5	22.27		23667	0.09

Totals			27372388	100.00
--------	--	--	----------	--------

MUMBAI
HPLC Report

Data File: D:\HPLC3\RA-NHPI\Result\NHPI-02Jul2024-ON\RA-NHPI-8-Crude-02Jul2024.dat
Method: D:\HPLC3\RA-NHPI\Method\RA-NHPI-01.met
Sample I.D: RA-NHPI-8-Crude-02Jul2024
Acquired: 7/2/2024 9:05:40 PM (GMT +05:30)
Printed: 7/3/2024 11:00:29 AM (GMT +05:30)
Instrument ID: HPLC3
Inj vol: 2ul
Vial No.: 46

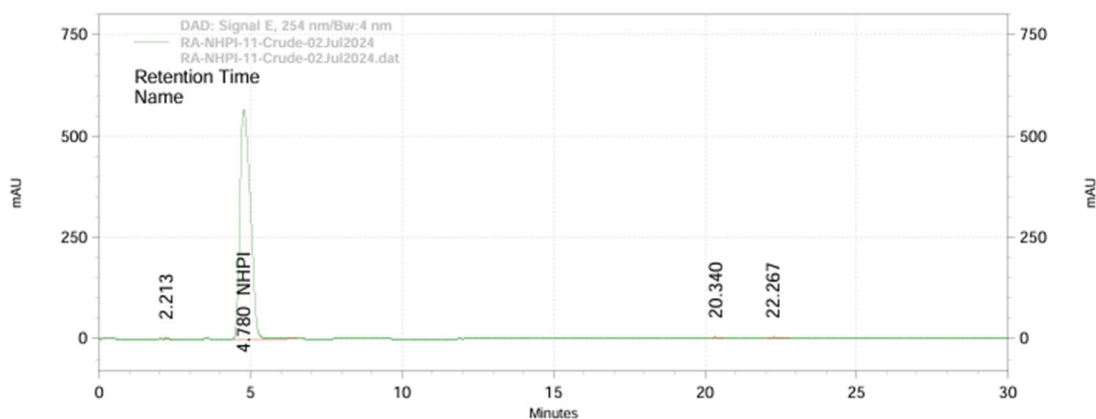


DAD: Signal E, 254
nm/Bw:4 nm Results

Pk #	R T	Name	Area	Area %
1	2.22		33502	0.10
2	4.79	NHPI	34406758	99.75
3	20.34		29415	0.09
4	22.27		24264	0.07
Totals			34493939	100.00

**MUMBAI
HPLC Report**

Data File: D:\HPLC3\RA-NHPI\Result\NHPI-02Jul2024-ON\RA-NHPI-11-Crude-02Jul2024.dat
Method: D:\HPLC3\RA-NHPI\Method\RA-NHPI-01.met
Sample I.D: RA-NHPI-11-Crude-02Jul2024
Acquired: 7/2/2024 9:42:42 PM (GMT +05:30)
Printed: 7/3/2024 11:01:07 AM (GMT +05:30)
Instrument ID: HPLC3
Inj vol: 2ul
Vial No.: 47



DAD: Signal E, 254
nm/Bw:4 nm Results

Pk #	R T	Name	Area	Area %
1	2.21		11360	0.04
2	4.78	NHPI	28183142	99.77
3	20.34		30052	0.11
4	22.27		24672	0.09
Totals			28249226	100.00

HPLC Results

SAMPLE NAME	HPLC AREA %
RA-PA-RC	95.31
RA-NHPI-CC-2	99.69
RA-NHPI-1+3-RC	99.81
RA-NHPI-05-CRUDE	99.60
RA-NHPI-08-CRUDE	99.75
RA-NHPI-11-CRUDE	99.77

SUMMARY

DATE	NAME	APPEARANCE	THEORITICAL AMOUNT (gms)	FORMED AMOUNT (gms)	YIELD %	MELTING POINT (°C)
05/06/24	RA- NHPI- 01	Yellow	1.6313	0.73	44.75	224
06/06/24	RA- NHPI- 02	Lime Yellow	1.6313	1.21	74.25	229.8
07/06/24	RA- NHPI- 03	Light Yellow	1.6313	0.747	45.79	223.2
10/06/24	RA- NHPI- 04	White	2.4958	0.1348	5.40	223
11/06/24	RA- NHPI- 05	White	4.8921	3.4558	70.64	228.8
12/06/24	RA- NHPI- 06	Light Yellow	6.07	4.26	70.14	233.3
14/06/24	RA- NHPI- 07	Yellow	1.6313	0.9028	55.3	229.8
17/06/24	RA- NHPI- 08	Light Yellow	3.2626	2.4796	76	228.7
17/06/24	RA- NHPI- 09	Orange	3.2626	1.2869	39.44	215
18/06/24	RA- NHPI- 10	Pale yellow	1.6313	1.14	69.88	226
20/06/24	RA- NHPI- 11	Light yellow	11.02	6 + 1.05	64	231

OBSERVATIONS & CONCLUSIONS:

1. Literature study was carried out.
2. Raw materials were identified and analyzed using Gas chromatography.
3. 11 experiments carried out by varying different parameters like,
 - a. Mole ratio of raw materials
 - b. Addition pattern
 - c. Different bases like NaOH and Na₂CO₃
 - d. Solvents like water, toluene and acetic acid
 - e. Cooking temperature
 - f. pH
4. The reaction was monitored using thin layer chromatography
5. The yield varied from 5 to 76%.
6. The experiment no. 8 gave the maximum yield among all the experiments which was 76%
7. The NHPI synthesized was purified using
 - a. Column chromatography
 - b. Recrystallization
8. The purity of synthesized NHPI was analyzed using HPLC by A%, and found to be +99%.
9. Two polymorphs were obtained namely white and yellow. The reaction at 60-65°C gave white polymorph where as yellow polymorph was obtained at 100°C reaction temperature.
10. Melting point was determined.
11. The final product was also confirmed by IR spectrophotometer and compared with the market sample.
12. TLC works in normal phase that was solvent used was non-polar but the column is polar.
13. HPLC that we used here works in reverse phase that is solvent used was polar and the C-18 column is non-polar.

ABBREVIATIONS:

1. NHPI: N-hydroxy phthalimide
2. RT: room temperature
3. RBF: Round bottom flask
4. PA: Phthalic anhydride
5. HAS: Hydroxyl ammonium sulfate
6. HAC: Hydroxyl ammonium chlorate
7. GC: Gas chromatography
8. HPLC: High Performance Liquid Chromatography
9. TLC: Thin Layer Chromatography
10. RM: Reaction mixture

BHAVNAGAR PLANT VISIT(24/06/24 to 28/06/24):

NAME	REMARK
BOILER	There was a centralised boiler facility that transported steam to the plant to meet their heating requirements. The boiler had a fluidised bed furnace with three beds, which had a pressure of -2mm WC to -5mm WC created by the ID Fan. Coal and fluidized material made of alumina and silica were used as fuel. The steam generated at full operation of the boiler had a pressure of 14Kg.
PRS	Known as the Pressure Reducing Station, steam came from the boiler at high temperature and couldn't be directly transported to the plant at that high pressure. So, the steam pressure was reduced to 9Kg, then transported to the plant for their use.
CHILLER	R134A was used as a refrigerant. In the industry, chilled water meant the temperature of the chiller water was 6°C. Normal water entered the heat exchanger from one side, and chilled refrigerant entered different tubes. The chilled water exited from the other side, and the refrigerant went to the condenser for cooling.
COOLING TOWER	It worked on the principle of humidification, in which warm/hot water was sprayed from the top of the tower, and the heat present in the water was taken by the air, causing the water coming down to cool. The cooled water in the industry had a temperature of 25-30°C.
COMPRESSOR	The rotary screw compressor compressed the air and made it oil, dust, and moisture-free.
ATFE	Known as an agitated thin film evaporator, it was used for short path distillation. It had a hot jacketed shell and a rotor assembly that rotated at high speed inside the shell. This was used in the MEE plant for waste treatment.
WFE	Known as a Wiped Film Evaporator, it was also used for short path distillation. It was used in the CTZ plant and designed to continuously separate volatile compounds by introducing a mechanically agitated thin film of feed material to a heated surface.
AOD Pump	The air-operated diaphragm pump was a pneumatic pump used for pumping material by creating suction with the help of air.
DOME FILTER	They were normal filters fitted with different filter cloths based on their mesh size, used to separate solids from a mixture of solids and liquids. Coating of ETFE (Ethyl tetrafluoroethylene) is done for acidic media to prevent SS from Corrosion.
ANFD	Known as an Agitated Nutsche Filter Dryer (ANFD), it was also known as a smart filter because it performed filtration along with drying. It contained an S-shaped stirrer hollow inside, through which vacuum traveled, ensuring uniform heating throughout the mixture. ANFDs were used to separate solid materials from liquids such as suspensions or slurries. The liquid was removed through the filter cloth while the solids were retained on top of it. The agitator helped improve the separation process by preventing compaction of solids on the filter cloth.
SIGMA AGITATOR	Used for mixing solids in gas and solids in solid, it was used in GT for the formulation of MERA 71.
RIBBON BLENDER	Used for mixing of solid and solid

JET MILLING	Used in the formulation process to make particles finer with the help of flowing high-pressure air, it couldn't be used when there was interference from air. It produced dry powder.
BEAD MILL	It was used to make particle sizes finer. In this process, powder was filled into a bead column and spun at high speed to achieve very fine particle sizes. This method was used for various formulations.
ACM	Known as an air classified mill, it was a type of grinding mill that used air to classify and transport particles. In this process, coarse particles fell back to the grinding zone for further grinding, while finer particles were carried upward by the air stream and escaped through an air outlet.
PLOUGH MIXER	Used to break lumps.
HOMOGENIZER	The ingredients were mixed or blended together to create a uniform consistency. This process broke down particles or liquids to create a homogeneous mixture, ideal for applications.
PPFRP TANK	In the CTZ plant, tanks that were used for storage of acidic water. They were lightweight and did not corrode with acid media. These tanks were made of polypropylene fiber-reinforced plastic, with the inner layer made of PP and the outer layer made of FRP for strength.
GRAPHITE HES	The heat exchanger, covered in graphite and arranged in a crossed tube manner, allowed one cooling media to travel through while cooling down vapors or materials from the other side. It was designed for corrosive materials, compact spaces, and environments with acidic media, adhering to Tubular exchange manufacturer association (TEMA) standards.
MECHANICAL SEALING	Mechanical seals were leakage control devices found on rotating equipment such as mixers. They prevented the leakage of liquids and gases from escaping into the environment.
SPARK TEST	This test was used to detect any cracks in the stainless steel glass-lined reactor (SSGL).
RVPD	It was known as a rotary vacuum paddle dryer, used for drying purposes in the CTZ plant.
CENTRIFUGAL PUMP	It worked on the Bernoulli equation and was designed to move fluid by transferring rotational energy from one or more driven rotors, called impellers.
PFD	Process flow diagram.
P&ID	Piping and instrumentation diagram.
STEAM TRAP	It was used to discharge condensate, air, and other incondensable gases from a steam system while preventing the escape of live steam.
PCF	The pillar centrifuge was a type of centrifuge used to separate solids in the GT plant.
BAROMETRIC HEIGHT	When designing a plant, for example, to achieve a specific pressure inside a reactor or storage tank, the height required is calculated using ρgh , where ρ is the density of the fluid, g is the acceleration due to gravity, and h is the height of the fluid column. For mercury to achieve a pressure equivalent to 1 atm, the height needed is 760 mm.
PLC	Programmable logic control was used when the number of controllers was low and for the control of manufacturing processes.
DCS	A distributed control system was used when the number of controllers was high.

PID	Proportional Integral Derivative controller was an instrument used by engineers to regulate temperature, flow, pressure, speed, and other process variables in industrial control systems.
HAZOP	Hazard and Operability study
VALVES	There were different types of valves used in a chemical industry depending on the need and type of material flowing through the valve. The globe valve was used to control the flow, and the ball valve and butterfly valve were used for large areas.
MEE	<p>A multiple effect evaporator, used in effluent treatment in a plant, operates by using the steam formed from the first column to heat the second column, and this process continues sequentially. They are used in two different ways:</p> <ol style="list-style-type: none"> 1. Forward feed, where the feed and steam (initially from the boiler) enter from the same side. 2. Backward feed, where the feed and steam (from the boiler) enter from different sides. <p>They are also used along with a Mechanical Vapour Recompressor (MVR).</p>
ETP	An effluent treatment plant, also known as the heart of a plant, is used to treat effluent before discharging it to sewage. Bio-enzymes are used for treatment in different tanks, aiming to reduce the Chemical Oxygen Demand (COD) to 250 ppm and the Biological Oxygen Demand (BOD) to 100 ppm in the effluent water.
SAND FILTER	Sand filters are used in the purification of treated water to remove suspended matter through a mechanism involving a bed made up of sand.
CHARCOAL FILTER	A charcoal filter is used to remove the smell from treated water through the phenomenon of adsorption.
PSA TOWER	Pressure swing adsorption tower is used to remove toxic gases through the phenomenon where, under high pressure, gases tend to be trapped onto solid surfaces, i.e., they are adsorbed.
SFD	Spin flash dryer
SPARKLER FILTER	It was used in the chemical industry. These filters helped remove solid impurities and unwanted particles from chemical solutions, ensuring product quality and consistency.

REFERENCES

- ⁱ Wikipedia
- ⁱⁱ MSDS of compounds from Thermo Fisher scientific
- ⁱⁱⁱ Chemical Book article
- ^{iv} COA from Merck
- ^v Wikipedia
- ^{vi} Patent number CN- 106831535A
- ^{vii} Through various experiments we got to know this solvent system is best for our use.
- ^{viii} Patent Number CN – 106831535A embodiment 5 with minor changes in addition pattern
- ^{ix} Patent Number CN – 106831535A embodiment 5 with minor changes in addition pattern of reactant and neutralisation of the acid form
- ^x The Journal of Physical Chemistry A 2007, 111, 2587-2601 – Experimental Section -C
- ^{xi} Patent Number CN – 106831535A embodiment 5 with minor changes in addition and cooking pattern
- ^{xii} Patent Number CN – 106831535A embodiment 5 with minor changes in addition pattern
- ^{xiii} Patent Number CN – 106831535A embodiment 5 with minor changes in addition pattern
- ^{xiv} Patent Number CN – 106831535A embodiment 5 with different addition pattern and amount used.
- ^{xv} The Journal of Physical Chemistry A 2007, 111, 2587-2601 – Experimental Section – B
- ^{xvi} Patent Number CN – 106831535A embodiment 5 with change in hydroxylamine salt and addition pattern
- ^{xvii} Sheldon, R. A., Arends, I. W. C. E., & ten Brink, G. J. (2002). Green Chemistry and Catalysis. Green Catalysis, pp. 107-109. This source details general methods for NHPI synthesis.
- ^{xviii} Liu, Y., & Yin, G. (2015). Metal-free Aerobic Oxidation Catalysis: N-Hydroxy phthalimide and Its Derivatives. Chemical Society Reviews, 44(15), 4605-4617. This source provides insights into the reaction conditions and yields.