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A combination of Isophthalonitrile, 5M Na hydrated oxide, and dioxane was placed in a round-bottom flask equipped with a magnetic follower. A reflux setup was so set up with the usage of a paraffin oil bath. The reflux was so stabilized at 90 oC and the mixture was allowed to reflux nightlong. Ammonia was liberated as a consequence of the condensation reaction. The absence of ammonium hydroxide from the adduct was so tested via TLC. Petroleum ether/ ethyl ethanoate ( 1: 1 ) was utilized as the nomadic stage and Isophthalonitrile was used as a mention.

No ammonium hydroxide was observed in the merchandise. The mixture was so rotary evaporated to take any dioxane residue. A white precipitate was formed. The white solid was so dissolved in H2O and acidified to pH 2 in an ice-bath by 4M hydrochloric acid. The merchandise ‘ s pH was so tested with litmus paper. A white precipitate was produced as the pH decreased. The merchandise was so vacuum filtered via Buchner filtration. The solid adduct was washed with H2O several times.

The pH was tested one time more to guarantee the sourness of the merchandise. The white solid was so oven dried at 85 oC overnight. A sample was so taken for proton NMR analysis to find the merchandise obtained. It was found that the merchandise was so isophthalic acid. Once out of the oven, the merchandise was refluxed in ethyl alcohol ( 50 cm3 ) at 50-60 oC for a period of three hours to divide Na chloride salt from isophthalic acid as the isophthalic acid is soluble in ethyl alcohol and the Na hydrated oxide is indissoluble in ethyl alcohol. The white mixture transformed into a light pink creamy composite.

Extra ethyl alcohol ( 10 cm3 ) was so transferred into the round-bottom flask ( 250cm3 ) and the mixture was left to reflux for farther 25 proceedingss. The solution was so hot filtrated. It was so washed down with ambient temperature ethyl alcohol. The liquid merchandise was so transferred into a round-bottom flask ( 250cm3 ) and was rotary evaporated to take extra ethyl alcohol. The concluding merchandise was so air-dried and weighed.

89. 23 % output was acquired. A sample was so taken for proton NMR ( Bruker ) , Infrared spectrometry ( Nicolet 100IR ) and mass spectral analysis.

The adduct was confirmed as pure isophthalic acid. [ 27 ]Scheme 7 ( eMolecule )Synthesis of Isophthalic acid from Isophthalonitrile. [ 27 ]

## Synthesis of Furan-2, 5-dicarboxylic acid

A mixture of 5-methyl-2-furonitrile ( 0. 5 g, 4. 67 mmol ) , 5M Na hydrated oxide ( 6 cm3 ) , and dioxane ( 3 cm3 ) was transferred into a round-bottom flask ( 250cm3 ) equipped with a magnetic follower. A reflux setup was so set up with the usage of paraffin oil bath.

The reflux was so stabilized at 90 oC and the mixture was allowed to reflux overnight. A xanthous mixture was formed. The setup was allowed to chill down to room temperature. The mixture was so rotary evaporated to take the volatile dioxane. The precipitate obtained was so dissolved in H2O and was acidified to pH 2 in an ice-bath by 4M hydrochloric acid. The acid was so left in the ice-bath to chill to ease crystal formation.

No crystals were observed. The sample was one time once more rotary evaporated. A xanthous solid remained in the flask. A sample of the merchandise was so sent for proton NMR and mass spectroscopy and was found to be the intermediate 5-methyl-2-furancarboxylic acid. The xanthous intermediate was so weighed and 86.

7 % output was obtained. The experiment was so terminated at the intermediate phase due to miss of laboratory clip. [ 27 ]Scheme 8 ( eMolecule )Synthesis of Furan-2, 5-dicarboxylic acid from 5-methyl-2-furonitrile. [ 27 ]

## Synthesis of 5-nitro-2-thiophene carboxylic acid

A mixture of 5-nitro-2-thiophene carbonitrile ( 0. 3 g, 1.

94 mmol ) , 5M Na hydrated oxide ( 5 cm3 ) , and H2O ( 3 cm3 ) was placed into a round-bottom flask ( 250cm3 ) equipped with a magnetic follower. A reflux setup was so set up with the usage of a paraffin oil bath. The reflux was so stabilized at 90 oC and the mixture was allowed to reflux overnight.

The mixture ‘ s colour had changed from xanthous to dark ruddy after 30 proceedingss of refluxing. The mixture was so allowed to chill down to room temperature. It was so acidified in an ice-bath by 4M hydrochloric acid. Fumes formed in the round-bottom flask as the acid was being added. The colour changed from dark ruddy to dark brown as the pH dropped. The mixture was so left to crystallise in an ice-bath for a period of one hr.

Dark brown pellets were formed. The merchandise was so centrifuged for 10 proceedingss at 4, 000 RPM ( MSE Henderson Blomedical LTD ) to divide the pellets from the liquid. The top liquid bed was discarded.

The pellets were so left to air-dry. They were so weighed and 50. 45 % output was obtained. A sample was so taken for proton NMR and mass spectral analysis. The merchandise was found to be 5-nitro-2-thiophene carboxylic acid. [ 27 ]Scheme 9 ( eMolecule )Synthesis of 5-nitro-2-thiophene carboxylic acid from 5-nitro-2-thiophene carbonitrile. [ 27 ]

## Synthesis of methyl pyrrole-2, 5-dicarboxylic acid

A mixture of 1, 5-dimethyl-2-pyrrole ( 0. 5 g, 4.

16 mmol ) , 5M Na hydrated oxide ( 5 cm3 ) , and H2O ( 3 cm3 ) was transferred into a round-bottom flask ( 250cm3 ) equipped with a magnetic follower. A reflux setup was so set up with the usage of a paraffin oil bath. The reflux was so stabilized at 90 oC and the mixture was allowed to reflux overnight. The setup was allowed to chill down to room temperature.

A clear solution was formed. The mixture was so acidified to pH 2 in an ice-bath by 4M hydrochloric acid. The clear solution turned into a white paste as the acid was being added. The paste was so vacuum filtered via Buchner filtration setup.

The merchandise was so oven dried at 80 oC overnight. The intermediate was so weighed and 70. 34 % output was obtained. A sample was taken for proton NMR and Mass spectroscopy. Both techniques failed to bring forth consequences due to the samples high unsolvability. An Infra-red spectrum was so obtained.

It identified the merchandise as the intermediate 1, 5-dimethyl pyrrole-2-caboxylic acid. [ 27 ]Scheme 10 ( eMolecule )Synthesis of methyl pyrrole-2, 5-dicarboxylic acid from 1, 5-dimethyl-2-pyrrole. [ 27 ]

## Consequences

## Merchandise name

## Product molecular expression

## Product molecular mass ( g/ mole )

## Theoretical Weight ( g )

## Experimental Weight ( g )

## Percentage Output ( % )

Isophthalic acidC8H6O4166. 130.

6500. 58089. 235-methyl-2-furan carboxylic acidC6H6O3126. 110. 7290. 58886. 75-nitro-2-thiophene carboxylic acidC5H3NO4S173. 140.

3370. 17050. 451, 5-dimethy pyrrole-2-carboxylic acidC7H9NO2139. 150. 7040. 58070. 34

## Table 1

Information & A ; Results of merchandises synthesized.

## Isophthalic Acid:

Figure 6 ( eMolecule )Structure of isophthalic acid with numbered H environments.

## Proton NMR

i?¤ : ( 1 ) 7. 59 ( 1H ) three, ( 2 ) ( 4 ) 8. 24 ( 3H ) doublet, ( 3 ) 8. 65 ( 2H ) vest

## Infrared spectrum

O-H group wide extremum at 3240-2440 cm-1, C= O group extremum at 1760-1640 cm-1, H-C= C group ( aromatic ) weak extremum at 3150-3000 cm-1, C= C group ( aromatic ) extremum at 1600-1450 cm-1.

## Mass spectrum

M+ 165. 88 m/z, M-1.

51 ( H ) 164. 37 m/z, M-15. 95 ( CH4 ) 149. 93 m/z, M-17. 05 ( OH ) 148.

83 m/z, M-28 ( CO ) 137. 88 m/z, M-45. 29 ( COA­2H ) 120.

59 m/z, M-89. 95 ( 2COA­2H ) 75. 93 m/z, M-90. 95 ( 2COA­2H, H ) 76. 93 m/z, M-100. 97 ( 2COA­2H, C ) 64.

91 m/z, M-114. 98 ( 2COA­2H, C2H2 ) 50. 90 m/z.

## 5-methyl-2-furan carboxylic acid

Figure 7 ( eMolecule )Structure of 5-methyl-2-furan carboxylic acid with numbered H environments.

## Proton NMR

i?¤ : ( 1 ) 2. 51 ( 3H ) vest, ( 2 ) 6.

29 ( 1H ) doublet, ( 3 ) 7. 11 ( 1H ) doublet, ( 4 ) 12 ( 1H ) vest.

## 5-nitro-2-thiophene carboxylic acid

Figure 8 ( eMolecule )Structure of 5-nitro-2-thiophene carboxylic acid with numbered H environments. Figure 9 ( eMolecule )Molecular construction of CD3OD ( Deuterated methyl alcohol ) with numbered H environments.

## Proton NMR

i?¤ : ( 1a ) 3. 30 ( 3H ) vest, ( 2a ) 4. 90 ( 1H ) vest.

## Mass spectrum

M+ 177. 20 m/z, M-44. 04 ( CO2 ) 133. 16 m/z, M-92. 20 ( CO2, NO2 ) 85.

02 m/z, M+44. 25 ( CO2 ) 221. 25 m/z, M+84.

27 ( 2CO2 ) 261. 27 m/z, M+128. 27 ( 3CO2 ) 305. 32 m/z, M+172. 27 ( 4CO2 ) 349. 37 m/z, M+216. 27 ( 5CO2 ) 393. 42 m/z, M+260 ( 6CO2 ) 437.

47 m/z.

## 1, 5-dimethyl pyrrole-2-carboxylic acid

Figure 10 ( eMolecule )Structure of 1, 5-dimethyl pyrrole-2-carboxylic acid with numbered H environments.

## Proton NMR

Not obtained

## Mass Spectrum

Not obtained

## Infrared spectrum

O-H group wide extremum at 3240-2440 cm-1, -CH3 group extremum at 3000-2800 cm-1, C= O group extremum at 1760-1640 cm-1.

## Conclusion & A ; Discussion

The Suitable fresh compounds synthesized were designed based on the constructions of L-glutamate and 2-oxoglutarate so as to move as possible glutamate dehydrogenase inhibitors forestalling the transition of L-glutamate into 2-oxoglutarate in the Plasmodium Falciparum parasite. The compounds were synthesized under alkalic conditions instead than acidic conditions due to the celerity and undemanding nature of the reaction. 4M Hydrochloric acid was prepared by the add-on of the acid onto the H2O. The add-on of H2O onto a concentrated acid causes release of a big measure of heat ( exothermal reaction ) . Therefore, when H2O is added onto an acid ; H2O turns into hot steam which so bursts out of the flask/container along with the scorching concentrated acerb eating the surface about it every bit good as doing terrible Burnss and hurts to the chemist managing it. Isophthalic acid was synthesized and obtained by the hydrolysis of the Isophthalonitrile ‘ s two cyano groups.

The compound was analyzed via proton NMR which showed signals for all expected H environments ; infrared which showed extremums of all accounted for functional groups every bit good as mass spectroscopy ( as explained in the consequences subdivision ) . It was concluded that the merchandise obtained was so isophthalic acid. However, a output of 89. 23 % was obtained. That might hold been a consequence of legion transportations of the chemical throughout the experimentation.

The synthesis of furan-2, 5-dicarboxylic acid from 5-methyl-2-furonitrile was non carried out. The experiment was stopped at the intermediate 5-methyl-2-furancarboxylic acid. Further oxidization had to be done to change over the methyl group into a carboxylic acid. The oxidization was meant to be preformed with the assistance of H2O and KMnO4 ( a strong oxidizing agent ) . [ 28 ] However, due to the deficiency of research lab Sessionss the oxidization of methyl was non achieved. A output of 86. 7 % of the intermediate was obtained. The compound was analyzed via proton NMR screening signals at all appropriate H environments every bit good as infrared spectrometry which indicated the accounted for functional groups.

It was concluded that the merchandise gained was so the intermediate 5-methyl furan-2-carboxylic acid. 5-nitro-2-thiophene carbonitrile was hydrolyzed under basic conditions to give 5-nitro-2-thiophene carboxylic acid. A merchandise was obtained from the experiment with a output of 50. 45 % . However the designation of the chemical was non possible. A proton NMR was obtained demoing merely solvent deuterated methyl alcohol ( cd3od ) extremums.

Mass spectrometric analysis was besides carried out. The mass spectrum informations showed a reiterating addition of 44 units, which is consistent with the addition of a CO2 group. As a decision, the chemical was believed to hold polymerized during the acidification of the carboxylate group into a carboxylic acid group. Further proving was required to turn out the polymerisation reaction, nevertheless due to the deficiency of laboratory clip ; the premise could non be confirmed.

-H2Where R = NO2, R’= CO2H( -R & A ; -R ‘ bonds have been shortened for lucidity ) . Scheme 11 ( eMolecule )The polymerisation reaction of thiophene. [ 6 ]Methyl pyrrole-2, 5-dicarboxylic acid was non acquired from pyrrole-2, 5-dicarboxylic acid. The experiment was eliminated at the intermediate 1, 5-dimethy pyrrole-2-carboxylic acid due to clip deficit. Further oxidization had to be done to change over the 5-methyl group into a carboxylic acid. The oxidization was meant to be preformed with the assistance of H2O and KMnO4 ( a strong oxidizing agent ) .

[ 28 ] A output of 70. 34 % of what was believed to be the intermediate was obtained. However the individuality of the compound remained unknown. An effort to analyse and authenticate the merchandise took topographic point but neither proton NMR nor a mass spectrum were obtained due to its high unsolvability in organic dissolvers. The compound was believed to hold polymerized during the acidification by 4M HCl of the 2-carboxylate group into a 2-carboxylic acid group. Further testing was necessary to corroborate the polymerisation reaction, nevertheless due to the deficiency of laboratory clip ; the premise could non be definite.-H2Where R = CO2H( -R & A ; -CH3 bonds have been shortened for lucidity ) . Scheme 12 ( eMolecule )The polymerisation reaction of pyrrole.

[ 6 ] [ 29 ]

## Mentions

Irwin W. Sherman, 1998, Malaria: parasite biological science pathogenesis and protection, 3-10, 140, ASM Press. Grant Lewison, Divya Srivastava. 2008, Malaria research, 1980-2004, and the load of disease, Acta Tropica, 106 ( 2 ) , 96-103. Arthur Israel Vogel, B. S. Furniss, 1989, Vogel ‘ s text edition of practical organic chemical science, 671, 1062-1063, 1228-1230, 1271-1272, Longman. I.

Safeukui, F. Mangou, D. Malvy, P. Vincendeau, D. Mossalayi, G.

Haumount, R. Vatam, P. Olliaro, P. Millet. 2004, Plasmodium berghei: dehydroepiandrosterone sulphate reverses chloroquino-resistance in experimental malaria infection ; correlativity with glucose 6-phosphate dehydrogenase and glutathione synthesis tract, Biomedical Pharmacology, 68 ( 10 ) , 1903-1910. Bruce Alberts, 1983, Molecular Biology of the Cell, Page 71, Garland Pub.

T. W Graham Solomons, Graig B. Fryhle, 2007, Organic Chemistry 9th edition, 79, 805-808, 451-454, Wiley. WHO, 2008, World Malaria study 2008, 9, World Health Organization.

John Daintith, 2007, Oxford Dictionary of Chemistry 6th edition, 344, Oxford University Press. N. S. Postma, E. C. Mommers. ( 1996 ) , Oxidative Stress in Malaria ; Implications for Prevention and Therapy, Pharmacy World & A ; Science, 18 ( 4 ) , 121-127.

Peter. B. Bloland, WHO, 2001, Drug Resistance in Malaria, 4, 1-3, World Health Organization. I. M. Aparicio, A. Marin-Menendez, A. Bell, P.

Engel. ( 2010 ) , Susceptibility of Plasmodium falciparum to glutamate dehydrogenase Inhibitors. A possible new antimalarial mark, Molecular & A ; Biochemical Parasitology, 172 ( 2 ) , 152. K. Becker, L. Tilley. ( 2004 ) , Oxidative Stress in Malarial Parasite-Infected Red blood cells: Host-Parasite Interactions, Internation Journal for Parasitology, 34 ( 2 ) , 164-165.

O. Erel, A. Kocyigit. ( 1997 ) , Oxidative Stress and Antioxidative Status of Plasma and Erythrocytes in Patients with Vivax Malaria, Clinical Biochemistry, 30 ( 8 ) , 631. Bernard A. Marcus, 2009, Malaria: Deadly Diseases & A ; Epidemics, 14-15, Infobase Publishing.

RSC, Malaria no more? , April 2008, hypertext transfer protocol: //www. rsc. org/chemistryworld/Issues/2008/April/MalariaNoMore.

asp, 26/03/2011, Online Source. Institute of Medicine ( U. S. ) , A S. C. Oaks, 1991, Malaria: obstructions and chances, 26, National Academies Press. X. Liang, W.

S. Fang, 2006, Medicinal chemical science of bioactive natural merchandises, 185, John Wiley and Sons. Caroline Hannaway, 2008, Biomedicine in the Twentieth Century: Practices, Policies, and Politicss, 291, IOS Press. J. Brent, 2005, Critical attention toxicology: diagnosing and direction of the critically poisoned patient, 674, Elsevier Health Sciences.

D. T. Okpako, 1991, Principles of pharmacological medicine: a tropical attack, 240, Cambridge University Press. T. K. Chatterjee, 2004, Chemotherapy of topical parasitic infections, 98, PHI Learning Pvt.

Ltd. Yunbo Song, 2011, Design Selective Glutamate Dehydrogenase Inhibitors? , 59, Lambert Academic Publishing AG & A ; Co KG. R. Sagar, 2009, Together with Chemistry: Lab Manual 2nd Edition, 188, Rachna Sagar Pvt. Ltd. N.

Kohli, 2009, Longman Science Chemistry 10, 49-50, Pearson Education India. M. A.

Fox, J. K. Whitesell. 2004, Organic Chemistry, 609, Jones & A ; Bartlett Learning. P. Lignier, J. Estager, N.

Kardos. 2011, Swift & A ; efi¬? cient sono-hydrolysis of cyanides to carboxylic acids under basic status: Function of the oxide anion group in the hydrolysis mechanism, Ultrasonics Sonochemistry, 18 ( 1 ) , 29. A. Milek, M. Sc thesis, 2008, Design & A ; synthesis of fresh GDH inhibitors as possible antimalarial agents, London Metropolitan University. F. Yang, J. Sun.

2004, Oxidation of methylbenzenes to benzoic acids by O in non-acidic dissolvers, Tetrahedron, 60 ( 5 ) , 1225-1226. George G. Odian, 2004, Principles of Polymerization 4th edition, 2-5, 9, John Wiley and Sons.