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# Synthesis of pyranopyrazole and pyranopyrimidine derivatives using magnesium oxide nanoparticles and evaluation as corrosion inhibitors for lubricants

**Ahmad R. Salih*** |**Zainab A. K. Al-Messri**

*Department of Chemistry, College of Science, University of Baghdad, Baghdad, Iraq*

***Corresponding Author:**
Zainab A. K. Al-Messri
Email:zainababdulzahra@sc.uobaghdad.edu.iq
Tel.: +009647702981597

A three-component, one pot reaction was used to synthesize different pyranopyrazole, tetrahydrochromene, and pyranopyrimidine derivatives using magnesium oxide nanoparticles with a faster reaction time, higher yield, and higher purity than a conventional piperidine base catalyst.
The prepared compounds were identified by FT-IR, $^{1}$HNMR, $^{13}$CNMR spectroscopy, and evaluated as good copper corrosion inhibitors by blending with a base stock lubricant provided by the Iraqi Midland Refineries Company/Al-Daura according to ASTM–D130.

**KEYWORDS**

Pyranopyrazole; tetrahydrochromene; pyranopyrimidine; lubricants; copper corrosion inhibitor.

## Introduction

Heterocyclic containing pyranopyrazoles, tetrahydrochromenes, or pyranopyrimidines moiety was found in bioactive natural and synthetic compounds, with unique properties such as, antimicrobial [1,2], anti-inflammatory [3,4], antitumor [5-7], antidiabetic [8], and antioxidant [9]. The synthesis of these interesting compounds was due to their significance. Therefore, several procedures were developed as multicomponent reactions (MCRs) using different catalysts [10-13].

Generally, pyranopyrazole, tetrahydrochromene, or pyranopyrimidine compounds are synthesized by three-component reactions of arylaldehydes and malononitrile with pyrazolinone, dimedone, barbituric acid, or Meldrum's acid respectively.

Pyranopyrazoles and pyranopyrimidines were synthesized using different catalysts, such as piperidine, triethyl amine, pyrrolidine, morpholine, piperazine [14], and Dibutylamine (DBA) [15], acid catalysts montmorillonite K-10 clay [16], and aluminum hexagonal mesoporous silica Al-HMS [17], amino acids, such as L-proline [18]. Several inorganics were also used as catalysts, such as potassium fluoride, sodium hydroxide, cesium carbonate, sodium carbonate, sodium bicarbonate, sodium phosphate [19], ammonium chloride [20], diammonium hydrogen phosphate (DAHP) [21] and Alum [22, 23]. Ionic liquids, triethylammonium hydrogen sulphate $[Et_3NH][HSO_4]$ [24], and $[(CH_2)_4SO_3HMIM][HSO_4]$,3-Methyl-1-(sulphonic acid) butyl-imidazolium hydrogen sulphate were used as catalysts [25].

The use of nanocatalysts in the synthesis of pyranopyrazoles increased intensely in the last decade. A variety of nano-sized catalysts were used, including magnesium oxide [26], magnetic $Fe_3O_4$ [27], titanium dioxide [28],

nanosilica [29], and nanoporous silica (SBA-Pr-$SO_3H$) [30].

In this work, we reported a one-pot with three component reaction to produce pyranopyrazole, tetrahydrochromene, and pyranopyrimidine compounds using nanosized magnesium oxide as an effective base catalyst.

## Experimental

*Materials and instruments*

All chemicals and solvents were supplied by Merck, Sigma-Aldrich, BDH, and CDH. The Stuart Scientific SMP1 apparatus is used to measure melting points. Infrared spectra were recorded using a Shimadzu FTIR-8400S Spectrophotometer, at the Department of Chemistry/College of Science/University of Baghdad.

$^1$H NMR and $^{13}$C NMR spectra were recorded on a Varian model ultra-shield at 500 MHz and 125 MHz, respectively, using DMSO-$d_6$ as a solvent and the chemical shifts were stated in part per million (ppm) relative to TMS as an internal reference, at Tehran University, Iran.

*Test methods*

Thin-layer chromatography (TLC) was performed on aluminum sheets pre-coated with silica gel 60 supplied by Merck. The eluent was a mixture of petroleum ether and ethyl acetate (6:4). Iodine vapor was used to detect spots. The American Society of Testing and Materials (ASTM/D130) was used to assess corrosion stability. A polished copper strip was dipped in the oil blend sample and heated at 100 °C for three hours. The tarnish level was measured against a standard of copper strip corrosion [31].

*Synthesis 2-(4-hydroxyl-3-methoxybenzylidine) malononitrile (1)*

A mixture of 4-hydroxyl-3-methoxy benzaldehyde (vanillin) (0.01mole, 1.5 g), malononitrile (0.01mole, 0.66 g) and two drops of pipridine or magnesium oxide nanoparticles (0.005mole, 0.2 g) in ethanol solvent (25 mL) was refluxed for an appropriate time. At the end of the reaction, the mixture was cooled, the precipitate was filtered, and recrystallized from ethanol.

*General procedures for synthesis (2-6)*

*A. Standard method*

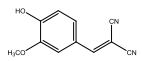
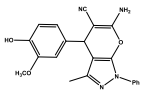
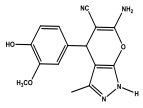
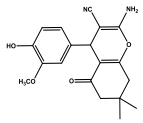
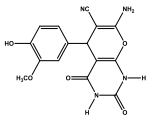
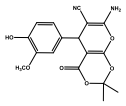
A mixture of 2 – (4 – hydroxyl – 3 – methoxybenzylidene malononitrile (1) (0.01 mole, 2 g) and 3 – methyl – 1 – phenyl – 2 – pyrazolin – 5 – one or 5-methyl-2,4-dihydro-3H-pyrazol-3-one or 5,5-dimethyl-1,3-cyclohexandion (dimedone) or pyrimidine-2,4,6 (1H, 3H, 5H) -trione (barbituric acid) or 2,2-dimethyl-1,3-dioxane-4,6-dione (Meldrum's acid), and two drops of pipridine or magnesium oxide nanoparticles (0.005 mole, 0.2 g) in ethanol (10 mL), was refluxed for an appropriate time. At the end of the reaction, the mixture was cooled, the formed precipitate was filtered, and recrystallized from ethanol[32].

*B. One-pot method*

A mixture of 4-hydroxyl-3-methoxy benzaldehyde (vanillin) (0.01mole, 1.5 g), malononitrile (0.01 mole, 0.66 g) and 3-methyl-1-phenyl-2-pyrazolin-5-one, 3-methyl-2-pyrazolin-5-one, 5,5-dimethyl-1,3-cyclohexandion (dimedone), pyrimidine -2,4,6-trione (barbituric acid), or 2,2-dimethyl-1,3-dioxane-4,6-dione (Meldrum's acid) and magnesium oxide nanoparticles (0.005 mole, 0.2 g) in ethanol (10 mL), was refluxed for an appropriate time. At the end of the reaction, the mixture was cooled, the formed precipitate was filtered, and recrystallized from ethanol[33].

Table 1 lists the physical properties of the prepared compounds (1-6).

**TABLE 1** Physical properties of the prepared compounds (1-6)

| *Comp.no* | Structure | Compound name | Molecular Weigh, g/mole | Color | m.p., °C | Pipridine Reaction time | Pipridine Yiel, % | Pipridine Rf. | MgO Reaction time | MgO Yield, % | MgO Rf. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 |  | 2-(4-hydroxy-3-methoxybenzylidene) malononitrile | 200.06 | Yellow | 134-136 | 1 hr. | 79.7 | 0.716 | 5 min. | 90.98 | 0.710 |
| 2 |  | 6-amino-4-(4-hydroxy-3-methoxyphenyl)-3-methyl-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile | 374.40 | Light Yellow | 142 | 1 hr. | 90.962 | 0.393 | 10 min. | 98.9 | 0.378 |
| 3 |  | 6-amino-4-(4-hydroxy-3-methoxyphenyl)-3-methyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile | 298.30 | Deep yellow | 232-234 | 2 hrs. | 90.66 | 0.317 | 10 min. | 96,33 | 0.253 |
| 4 |  | 2-amino-4-(4-hydroxy-3-methoxyphenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile | 340.38 | Off white | 228-230 | 3 hrs. | 93.46 | 0.593 | 10 min. | 94.89 | 0.609 |
| 5 |  | 7-amino-5-(4-hydroxy-3-methoxyphenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile | 328.28 | Deep Yellow | 150-153 | 3 hrs. | 18.33 | 0.435 | 5 min. | 56.29 | 0.451 |
| 6 |  | 7-amino-5-(4-hydroxy-3-methoxyphenyl)-2,2-dimethyl-4-oxo-4H,5H-pyrano[2,3-d][1,3]dioxine-6-carbonitrile | 344.32 | Yellow | 88-90 | 4 hrs. | 19.19 | 0.52 | 15 min. | 55.26 | 0.543 |

*Formulation of oil blends*

Blends of compounds (2-6) were prepared by dissolving and mixing 0.2% wt.\wt. of each compound in base oil 60 stock at 70-80 °C for 1 hour [34].

Base oil sixty stock was provided by the Iraqi Midland Refineries Company/Al-Daura. Table 2 contains a list of the oil's properties.

**TABLE 2** Properties of base oil/ 60 stock

| No. | Properties | Base stock 60 | Standard method |
|---|---|---|---|
| 1 | Specific gravity | 0.8842 | ASTM D-4052 |
| 2 | Viscosity Index(VI) | 106 | ASTM D- 2270 |
| 3 | Pour Point, °C | 0.0 | ASTM D-97 |
| 4 | Flash Point, °C | 242 | ASTM D-92 |
| 5 | Copper corrosion | 2a | ASTM D130 |
| 6 | Rust Preventing | Fail | ASTM D-665 |
| 7 | Color | 2.0 | ASTM D-1500 |

## Results and discussion

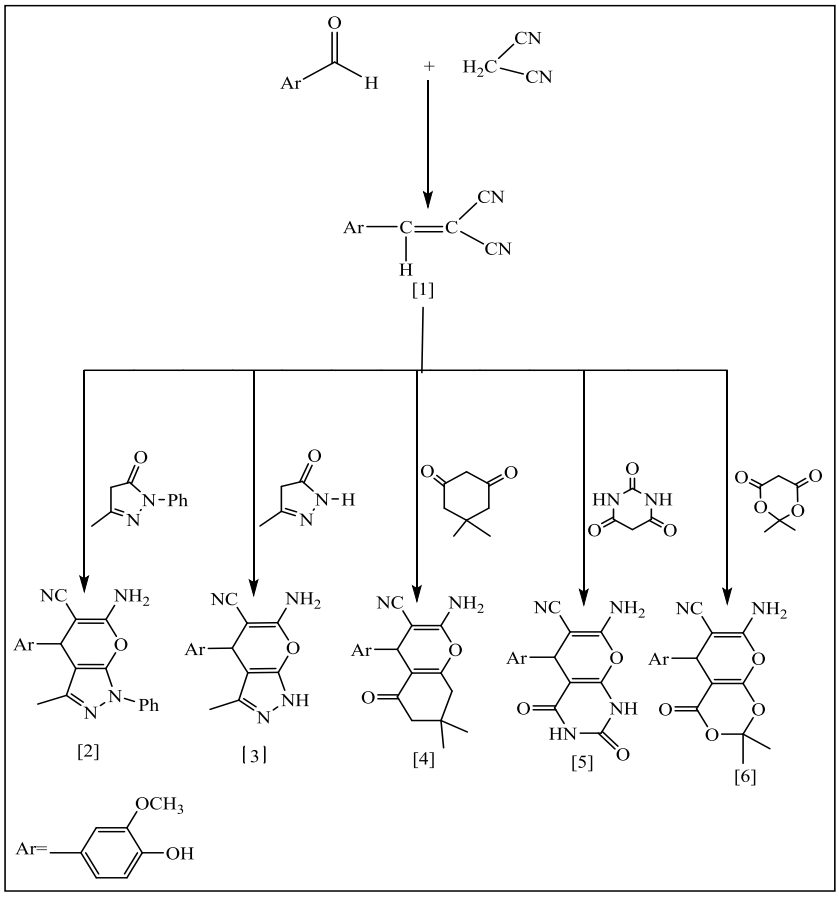
Different pyranopyrazole, tetrahydro-chromene, and pyranopyrimidine derivatives were synthesized using magnesium oxide nanoparticles in a three-component, one-pot reaction that had a faster reaction time, greater yields and higher purity than the conventional pipridine catalyst.

The Knoevenagel condensation of vanillin with malononitrile in the presence of magnesium oxide nanoparticles as a catalyst was used to synthesize 2-(4-hydroxyl-3-methoxybenzylidine) malononitrile (1), which reacted with 3-methyl-1-phenyl-2-pyrazolin-5-one, 3-methyl-2-pyrazolin-5-one, dimedon, barbituric acid, or Meldrum's acid according to the Michael addition, to afford (2-6). All these reactions and the formation mechanism [26] are shown in Schemes 1and 2.

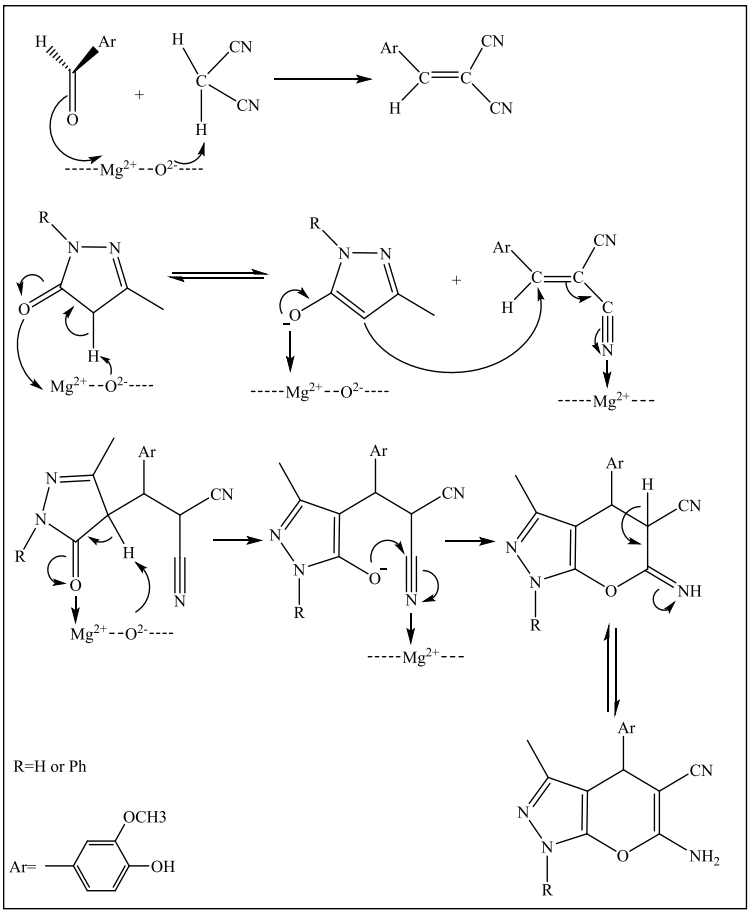
The structures of the prepared compounds (1-6) were proven using spectral data from FT-IR, $^{1}$HNMR, and $^{13}$CNMR analysis as illustrated in Tables 3, 4, and 5.

The prepared compounds (2-6) were evaluated for their anti-corrosion, and anti-rust activities by mixing, and blending 0.2% weight/weight of each compound with base stock according to ASTM–D130, and ASTM-D665, respectively.

The ASTM–D130 test method involved dipping a polished copper strip in the blend oil and heating it at 100°C for three hours. When the heating time was over, the strip was removed, cleaned and the tarnish level was considered against the standard of copper strip corrosion [35]. The prepared derivatives were found to be good anticorrosion lubricating oil additives, causing slight tarnish (1b) as compared with moderate tarnish (2a) on the base oil.



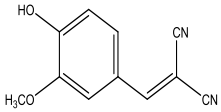
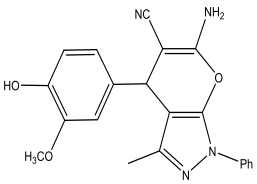

**SCHEME 1** Synthetic rout of compounds (2-6)




**SCHEME 2** Formation mechanism using MgO nanoparticles as catalyst

The ASTM-D665 rust-preventing test entails immersing a polished steel rod in a sample of blended oil mixed with water and heated at 60°C for four hours. The test rod was then examined for signs of rust. This test was run in duplicate, and to be considered successful, both test rods must be rust-free. The blends of compounds (2, 3, and 5) passed the rust-preventing test, while 2-amino-4-(4-hydroxy-3-methoxyphenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4), and 7-amino-5-(4-hydroxy-3-methoxyphenyl)-2,2-dimethyl-4-oxo-4H,5H-pyrano[2,3-d][1,3]dioxine-6-carbonitrile (6) failed.

**TABLE 3** FT-IR spectral data ($cm^{-1}$) of compounds (1-6)

| no. | Structure | νOH | $νNH_2$ | νCH arom. | νCH aliph. | ν C≡N | νC=C aliph. | νC=C arom. | δ (o-o-p) | Others |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 |  | 3427 | - | 3026 | 2956 | 2223 | 1604 | 1573<br>1508 | Tri-subs.<br>858, 815 | – |
| 2 |  | 3436 | Asym. 3373<br>Sym. 3321 | 3082 | 2947 | 2187 | 1595 | 1515<br>1490 | Mono-subs. 750 ,684<br>Tri-subs. 877, 806 | νC=N 1652 |

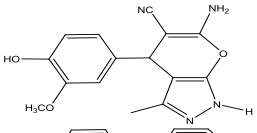
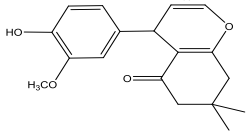
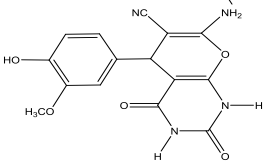
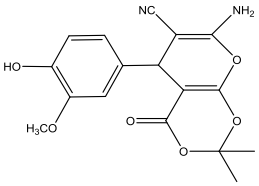
| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 3 |  | 3490 | Asym. 3326 Sym. 3274 | 3028 | 2979 | 2194 | 1604 | 1512 1485 | Tri-subs 879, 808 | νN-H 3218 νC=N 1656 |
| 4 |  | 3496 | Asym. 3404 Sym. 3323 | 3018 | 2962 | 2192 | 1654 | 1514 1465 | Tri-subs. 867, 819 | νC=O 1697 |
| 5 |  | 3419 | Asym. 3296 Sym. 3217 | 3029 | 2975 | 2225 | 1598 | 1514 1461 | Tri-subs. 869, 812 | νNH 3402 νC=O 1699 |
| 6 |  | 3440 | Asym. 3240 Sym. 3213 | 3213 | 2981 | 2221 | 1608 | 1517 1463 | Tri-subs. 856, 819 | νC=O 1728 |

**TABLE 4** $^{1}$HNMR spectral data (δ ppm) of compounds (1-6)

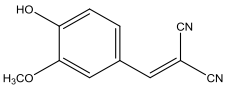
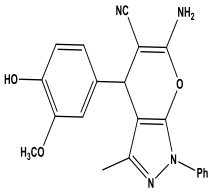
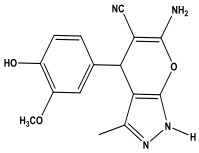
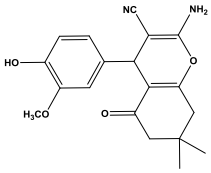
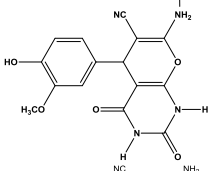
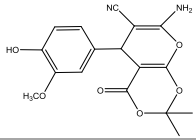
| Comp no. | Structure | O-H singlet | $NH_2$ singlet | C-H Aromatic multiplet | C-H Pyran singlet | $OCH_3$ (3H) singlet | $CH_3$ (3H) singlet | Other |
|---|---|---|---|---|---|---|---|---|
| **1** |  | 10.77 | _ | 7.61_ 6.96 | _ | 3.78 | _ | 1H(vinylic) Singlet 8.25 |
| **2** |  | 8.88 | 7.11 | 7.78-6.60 | 4.56 | 3.72 | 1.81 | _ |
| **3** |  | 8.82 | 6.52 | 6.77 - 6.52 | 4.48 | 3.69 | 1.81 | 1H(NH) Singlet 12.03 |
| **4** |  | 8.13 | 6.89 | 8.12-6.49 | 4.06 | 3.55 | 1.04 , 1.02 | 2H($CH_2$) Singlet 2.53 , 2.11 |
| **5** |  | 8.80 | 7.36 | 7.58-6.00 | 4.36 | 3.69 | _ | 1H(NH) Singlet 9.29 |
| **6** |  | 10.82 | 8.245 | 7.64-6.98 | 3.85 | 3.786 | 1.72, 1.48 | _ |

**TABLE 5** $^{13}$CNMR spectra data (δ ppm) of compounds (1- 6)

| No. | C-O-C | C=N pyrazol | C-O Phenol | CN | Arom. | O-$CH_3$ | C-C Pyran | $CH_3$ | Others |
|---|---|---|---|---|---|---|---|---|---|
| **1** | _ | _ | 148.60 | 113.72 | 154.50<br>128.41<br>123.37<br>116.68<br>115.00 | 56.20 | _ | _ | C=**C**-CN<br>75.61 |
| **2** | 159.69<br>,<br>147.85 | 145.94 | 145.84 | 120.28 | 145.94<br>138.05<br>134.98<br>129.75<br>126.49<br>120.57<br>115.96<br>112.40 | 56.14 | 36.83 | 13.13 | _ |
| **3** | 161.12<br>,155.1<br>6 | 147.76 | 145.67 | 120.20 | 136.02<br>135.82<br>121.35<br>115.85<br>112.04 | 56.05 | 36.29 | 10.29 | _ |
| **4** | 162.58<br>,158.7<br>9 | _ | 145.69 | 119.81 | 147.67<br>136.25<br>120.31<br>115.77<br>113.47<br>111.86 | 56.02 | 35.42 | 28.97<br>,27.07 | (C=O)<br>196.12<br>$CH_2$<br>50.48,<br>32.20 |
| **5** | 161.65<br>,157.6<br>1 | | 155.29 | 120.72 | 147.61<br>138.75<br>133.97<br>120.72<br>114.80<br>112.42 | 55.63 | 37.07 | _ | (C=O)<br>165.61 |
| **6** | 159.20<br>,154.3<br>2 | _ | 148.35 | 116.59 | 128.16<br>123.54<br>115.56<br>114.79<br>113.52 | 55.97 | 39.10<br>9 | _ | 2$CH_3$<br>17.51,<br>18.02<br>C=O<br>160.99 |

## Conclusion

Pyranopyrazole, tetrahydrochromene, and pyranopyrimidine derivatives were successfully prepared using pipridine or magnesium oxide nanoparticles as catalyst in a three-component, one-pot reaction of vanillin and malonitrile with pyrazolinone, dimedone, barbituric acid, or Meldrum's acid, respectively.

Magnesium oxide nanoparticles as a base catalyst was found to give faster reaction time, higher yield, and higher purity than a conventional piperidine.

The prepared compounds were evaluated as copper corrosion inhibitors, and anti-rust for lubricants by blending 0.2% with 60 stock base oil. Blends showed high corrosion resistance and good anti-rust properties.

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***Orcid:***
*Zainab A. K. Al-Messri:*
*https://www.orcid.org/0000-0002-7698-5884*

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