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A Novel Synthesis for New Substituted Imidazoles

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This paper is dedicated to Professor Habib Firouzabadi on the occasion of his honourable retirement and for his continous, past and future devotions and endeavours to the field of organic chemistry.MMH owes him a personal debt of deep gartitude for his encourgments and friendships.

The addition of dimethyl acethylendicarboxylate (DMAD) to thiosemicarbazone and guanylhydrazone of aldehydes afforded compoundes which were characterized to be new substituted imidazoles. Their structures were determined by ^{1}H NMR, IR, mass spectra and elemental analyses.

Keywords: Dimethyl acethylendicarboxylate**,** Thiosemicarbazone**,** Guanylhydrazone, Imidazole

INTRODUCTION

Imidazole functional group plays important roles in numerous bioactive compounds [1]. The pharmacological interest of the imidazole ring has been established, Nitroimidazoles being extensively used in therapy against amoebic, trichomonal, giardial and anaerobic infections, or as hypoxic cell radiosensitizers [2]. Metronidazole and substituted imidazoles (ketoconazole, fluconazole and itraconazole) are well-tolerated drugs that are potentially active against Leishmania, but their use in the treatment of cutaneous and visceral leishmaniasis has produced conflicting results [3]. Substituted imidazoles have for years been targets for the search for compounds that can modulate e.g. blood pressure, heart rate, CNS diseases, and drugs like clonidine and phentolamine have been marketed for the treatment of hypertension [4- 6].

Recently, the interest in this heterocyclic system has widened as it is a precursor to a class compounds, called room temperature ionic liquids [7]. They have become ubiquitous ligands in organometallic chemistry and catalysis [8-11]. These compounds are an important class of nitrogen-containing heterocycles and they constitute useful intermediates in organic synthesis [12-14]. Different synthetic strategies for this nucleus are well documented and described in the literature,[15].

We are interested in the chemistry of heterocyclic compounds containing nitrogen and sulfur atoms [16]. As a part of a research program on the synthesis of heterocyclic system containing nitrogen and sulfur in solvent system and

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with a view to extending the synthetic utility of dimethyl acetylendicarboxylate [17], we have investigated the addition of the latter to thiosemicarbazone and guanylhydrazone in methanol.

EXPERIMENTAL

The melting points were obtained using an Electrothermal IA 9100 Digital Melting Point apparatus. The ir spectra were recorded on a Bruker (400-4000 cm^{-1}) spectrometer. nmr spectra were recorded on a 300 MHz spectrometer using TMS as internal standard. Mass spectrometric measurements were made on an Agilent Technologies 6890 N Network GC system.

Synthesis of imidazoles

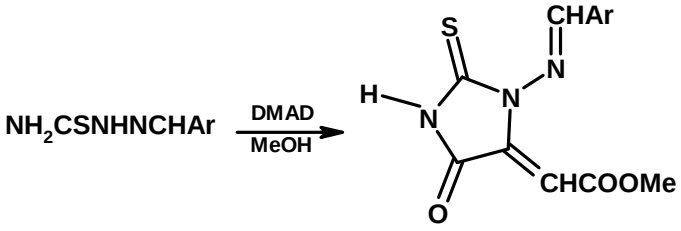
General procedure. A solution of thiosemicarbazone or guanylhydrazone (1 mmol) and DMAD (1 mmol) in 10 mL of MeOH was heated at reflux for 20 min . the solution was cold and the crystales that separated were collected.

1-(Benzylidene amino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one (1a).

benzaldehyde thiosemicarbazone and DMAD, this compound was obtained as yellow powder, Yield 93%, mp 285-286 ^oC; IR: SH 2760, CO 1725, 1699 cm^{-1}; ^1H nmr (300 MHz, DMSO-d_6) δ 3.67 (s, 3H, OMe), 6.59 (s, 1H, C=CH), 7.38 (m, 3H, Ar-H), 7.71 (m, 2H, Ar-H), 8.42 (s, 1H, N=CH), 12.81 ppm (br, 1H, NH); MS: m/z 289 (M^+); Anal. Calcd for $C_{13}H_{11}N_3O_3S$: C, 53.98; H, 3.80; N, 14.53. Found: C, 53.87; H, 3.52; N, 14.12.

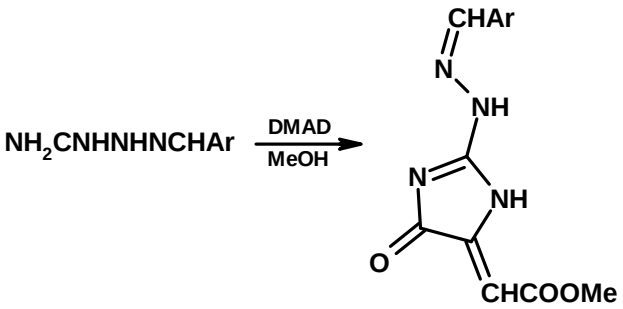
1-(*p*-Methylbenzylidene amino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one (1b).

p-methylbenzaldehyde thiosemicarbazone and DMAD, this compound was obtained as orang powder, Yield 92%, mp 281-283 ^oC; IR: SH 2764, CO 1731, 1701 cm^{-1}; ^1H nmr (300 MHz, DMSO- d_6) δ 3.19 (s, 3H, CH_3), 3.93 (s, 3H, OMe), 6.81 (s, 1H, C=CH), 7.45 (d, 2H, *J* = 7.0 Hz, Ar-H), 7.85 (d, 2H, *J* = 7.0 Hz, Ar-H), 8.63 (s, 1H, N=CH), 13.04 ppm (br, 1H, NH); MS: m/z 303 (M^+); Anal. Calcd for $C_{14}H_{13}N_3O_3S$: C, 55.45; H, 4.29; N, 13.86. Found: C, 55.70; H, 4.53; N 13.92.



Ar= 1a **C_6H_5-** e **m-$NO_2C_6H_4$-**
b **m-MeC_6H_4-** f **p-$NO_2C_6H_4$-**
c **C_6H_5-CH=CH-** g **2-pyrrol**
d **o-OHC_6H_4-** h **2-Furan**

Scheme 1



Ar= 2a **C_6H_5-** e **m-$NO_2C_6H_4$-**
b **m-MeC_6H_4-** f **p-$NO_2C_6H_4$-**
c **C_6H_5-CH=CH-** g **p-ClC_6H_4-**
d **o-OHC_6H_4-**

Scheme 2

1-(3-Phenyl-2-propenylidene amino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one (1c).

cinnamaldehyde thiosemicarbazone and DMAD, this compound was obtained as orange powder**,** Yield 98%, mp 287-289 ^{o}C; IR: SH 2768, CO 1728, 1692 cm^{-1}; ^{1}H nmr (300 MHz, DMSO- d_6) δ 3.88 (s, 3H, CH_3), 6.81 (s, 1H, C=CH), 7.31 (dd, 1H, *J* = 16.0, 9.0 Hz, =CH), 7.42 (d, 1H, *J* = 16.0 Hz, =CH), 7.57 (m, 3H, Ar-H), 7.82 (d, 2H, *J* = 7.0 Hz, Ar-H), 8.46 (d, 1H, *J* = 9.0 Hz, N=CH), 13.01 ppm (br, 1H, NH); ^{13}C nmr: δ 52.43, 114.23, 124.89, 127.54, 128.87, 129.46, 135.54, 142.78, 142.99, 160.97, 165.82 ppm; MS: m/z 315 (M^+); Anal. Calcd for $C_{15}H_{13}N_3O_3S$: C, 57.14; H, 4.12; N, 13.33. Found: C, 56.88; H, 4.10; N, 13.28.

1-(*o*-Hydroxybenzylidene amino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one (1d).

o-hydroxybenzaldehyde thiosemicarbazone and DMAD, this compound was obtained as yellow powder, Yield 91%, mp > 300 ^{o}C; IR: SH 2779, CO 1723, 1703 cm^{-1}; ^{1}H nmr (DMSO- d_6) δ 3.95 (s, 3H, OMe), 6.92 (s, 1H, C=CH), 7.22 (d, 1H, *J* = 8.0 Hz, Ar-H), 7.59 (m, 1H, Ar-H), 7.93 (d, 1H, *J* = 7.0 Hz, Ar-H), 8.97 (s, 1H, N=CH), 10.79 (s, 1H, OH), 13.19 ppm (br, 1H, NH); MS: m/z 305 (M^+); Anal. Calcd for $C_{13}H_{11}N_3O_4S$: C, 51.14; H, 3.63; N, 13.77. Found: C, 51.4; H, 3.80; N, 13.75.

1-(*m*-Nitrobenzylidene amino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one (1e).

m-nitrobenzaldehyde thiosemicarbazone and DMAD, this compound was obtained as orange powder, Yield 90%, mp > 300 ^{o}C; IR: SH 2768, CO 1721, 1702 cm^{-1}; ^{1}H nmr (300 MHz, DMSO- d_6) δ 3.68 (s, 3H, OMe), 6.71 (s, 1H, C=CH), 7.78 (dd, 1H, *J* = 7.0, 7.0 Hz, Ar-H), 8.18 (d, 1H, *J* = 7.0 Hz, Ar-H), 8.47 (dd, 1H, *J* = 7.0, 2.0 Hz, Ar-H), 8.62 (d, 1H, *J* = 2.0 Hz, Ar-H), 8.70 (s, 1H, N=CH), 12.80 ppm (br, 1H, NH); MS: m/z 334 (M^+); Anal. Calcd for $C_{13}H_{10}N_4O_5S$: C, 46.71; H, 2.99; N, 16.77. Found: C, 46.65; H, 2.87; N, 16.69.

1-(*p*-Nitrobenzylidene amino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one (1f).

p-nitrobenzaldehyde thiosemicarbazone and DMAD, this compound was obtained as yellow powder, Yield 93%, mp > 300 ^{o}C; IR: SH 2770, CO 1723, 1708 cm^{-1}; ^{1}H nmr (300 MHz, DMSO- d_6) δ 3.84 (s, 3H, OMe), 6.74 (s, 1H, C=CH), 8.11 (d, 2H, *J* = 8.0 Hz, Ar-H), 8.37 (d, 2H, *J* = 8.0 Hz, Ar-H), 8.72 (s, 1H, N=CH), 13.11 ppm (br, 1H, NH); MS: m/z 334 (M^+); Anal. Calcd for $C_{13}H_{10}N_4O_5S$: C, 46.71; H, 2.99; N, 16.77. Found: C, 46.70; H, 2.95; N, 16.74.

1-(2-Pyrrolmethylidene amino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one (1g).

2-pyrrolaldehyde thiosemicarbazone and DMAD, this compound was obtained as orange powder, Yield 92%, mp 258-260 ^{o}C; IR: NH 3327, SH 2760, CO 1695, 1652 cm^{-1}; ^{1}H nmr (300 MHz, DMSO- d_6) δ 3.62 (s, 3H, OMe), 6.03 (dd, 1H, *J* = 4.0, 7.0 Hz, Ar-H), 6.47 (d, 2H, *J* = 7.0 Hz, Ar-H), 6.87 (s, 1H, C=CH), 8.10 (s, 1H, N=CH), 11.41 (s, 1H, NH), 12.47 ppm (br, 1H, NH); MS: m/z 278 (M^+); Anal. Calcd for $C_{11}H_{10}N_4O_3S$: C, 47.48; H, 3.60; N, 20.14. Found: C, 47.7; H, 3.95; N, 20.72.

1-(2-Furanmethylidene amino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one (1h).

Furfural thiosemicarbazone and DMAD, this compound was obtained as orange powder, Yield 93%, mp 285-286 ^{o}C; IR: SH 2765, CO 1723, 1698 cm^{-1}; ^{1}H nmr (300 MHz, DMSO- d_6) δ 3.76 (s, 3H, OMe), 6.23 (dd, 1H, *J* = 4.0, 7.0 Hz, Ar-H), 6.67 (d, 2H, *J* = 7.0 Hz, Ar-H), 6.89 (s, 1H, C=CH), 8.21 (s, 1H, N=CH), 11.01 ppm (s, 1H, NH); MS: m/z 279 (M^+); Anal. Calcd for $C_{11}H_9N_3O_4S$: C, 47.31; H, 3.22; N, 15.05. Found: C, 46.96; H, 3.11; N, 14.84.

2-Yl-(benzyliden hydrazino)-3(H)-4-(methoxy carbonyl methylene)-imidazol-5-one (2a).

Benzaldehyde guanylhydrazone and DMAD, this compound was obtained as orange powder, Yield 92%, mp 218-220 ^{o}C; IR: NH 3436, 2700-3200, CO 1739, 1689 cm^{-1}; ^{1}H nmr δ 3.53 (s, 3H, OMe) 5.80 (s, 1H, C=CH), 7.51 (m, 3H, Ar-H), 7.88 (m, 2H, Ar-H), 8.15 (s, 1H, N=CH), 8.47 (br, 1H, NH), 9.21 (br, 1H, NH); ^{13}C nmr (300 MHz, DMSO-d_6) δ 52.40, 96.76, 129.46, 132.24, 132.35, 137.08, 161.88, 165.29, 168.26, 174.36; MS: m/z 272 (M^+); Anal. Calcd for $C_{13}H_{12}N_4O_3$: C, 57.73; H, 4.41; N, 20.58. Found: C, 57.32; H, 4.51; N, 20.82.

2-Yl-(p-methylbenzyliden hydrazino)-3(H)-4-(methoxy carbonyl methylene)-imidazol-5-one (2b).

p-methylbenzaldehyde guanylhydrazone and DMAD, this compound was obtained as yellow powder, Yield 85%**,** mp 180-182 ^{o}C; IR: NH 3438, 2800-3200, CO 1699 cm^{-1}; ^{1}H nmr

(300 MHz, DMSO-d_6) δ 2.37 (s, 3H, Me), 3.50 (s, 3H, OMe), 7.30 (d, 2H, *J* = 7.0 Hz, Ar-H), 7.78 (d, 2H, *J* = 7.0 Hz, Ar-H), 8.15 (s, 1H, N=CH), 8.92 (br, 1H, NH); MS: m/z 286 (M^+); Anal. Calcd for $C_{14}H_{14}N_4O_3$: C, 58.74; H, 4.89; N, 19.58. Found: C, 58.70; H, 4.53; N, 19.02.

2-Yl-(3-phenyl-2-propenyliden hydrazino)-3(H)-4-(methoxy carbonyl methylene)-imidazol-5-one (2c).

Cinnamaldehyde guanylhydrazone and DMAD, this compound was obtained as yellow powder, Yield 81%**,** mp 205-206 ^oC; IR: NH 3423 , 2800-3050 , CO 1739, 1690 cm^{-1}; ^1H nmr (300 MHz, DMSO-d_6) δ 8.70 (br, 1H, NH), 8.49 (br, 1H, NH), 8.03 (d, 1H, *J* = 9.0 Hz, N=CH), 7.62 (m, 2H, Ar-H), 7.41 (m, 3H, Ar-H), 7.31 (d, 1H, *J* = 16.0 Hz, C=CH), 7.03 (dd, 1H, *J* = 9.0, 16.0 Hz, HC=C), 5.73 (s, 1H, =CH exo methylen), 3.6 (s, 3H, OMe); MS: m/z 298 (M^+); Anal. Calcd for $C_{15}H_{14}N_4O_3$: C, 60.40; H, 4.69; N, 18.79. Found: C, 60.06; H, 4.25; N, 18.92.

2-Yl-(*o*-hydroxybenzyliden hydrazino)-3(H)-4-(methoxy carbonyl methylene)-imidazol-5-one (2d).

o-hydroxybenzaldehyde guanylhydrazone and DMAD, this compound was obtained as yellow powder, Yield 78%, mp 165-166 ^oC; IR: NH 3360, OH 2700-3600, CO 1730 cm^{-1}; ^1H nmr (300 MHz, DMSO-d_6) δ 3.66 (s, 3H, OMe), 5.78 (s, 1H, =CH exo methylen), 6.90 (m, 3H, Ar-H), 7.35 (m, 2H, Ar-H), 8.29 (s, 1H, N=CH), 8.45 (br, 1H, NH), 9.17 (br, 1H, NH), 10.17 (s, 1H, OH); MS: m/z 288 (M^+); Anal. Calcd for $C_{13}H_{12}N_4O_4$: C, 54.16; H, 4.17; N, 19.44. Found: C, 54.65; H, 4.07; N, 19.98.

2-Yl-(*m*-nitrobenzyliden hydrazino)-3(H)-4-(methoxy carbonyl methylene)-imidazol-5-one (2e).

m-nitrobenzaldehyde guanylhydrazone and DMAD, this compound was obtained as orange powder, Yield 76%**,** mp 247-248 ^oC; IR: NH 3411, 3120, CO 1702 cm^{-1}; ^1H nmr (300 MHz, DMSO- d_6) δ 3.57 (s, 3H, OMe), 5.86 (s, 1H, C=CH), 7.76 (dd, 1H, *J* = 8.0, 8.0 Hz, Ar-H), 8.23 (s, 1H, Ar-H), 8.31 (dd, 2H, *J* = 2.0, 8.0 Hz, Ar-H), 8.75 (br, 1H, NH), 8.81 (s, 1H, N=CH), 9.29 (br, NH); MS: m/z 317 (M^+); Anal. Calcd for $C_{13}H_{11}N_5O_5$: C, 49.21; H, 3.47; N, 22.08. Found: C, 48.87; H, 3.59; N, 21.92.

2-Yl-(*p*-nitrobenzyliden hydrazino)-3(H)-4-(methoxy carbonyl methylene)-imidazol-5-one (2f).

p-nitrobenzaldehyde guanylhydrazone and DMAD, this compound was obtained as orange powder, Yield 88%, mp 198-220 ^oC, IR: NH 3389, 3130, CO 1718 cm^{-1}; ^1H nmr (300 MHz, DMSO-d_6) δ 3.62 (s, 3H, OMe), 5.88 (s, 1H, C=CH), 8.15 (s, 1H, N=CH), 8.19 (d, 2H, *J* = 8.0 Hz, Ar-H), 8.31 (d, 2H, *J* = 8.0 Hz, Ar-H), 8.72 (br, NH), 9.31 (br, NH); MS: m/z 317 (M^+); Anal. Calcd for $C_{13}H_{11}N_5O_5$: C, 49.21; H, 3.47; N, 22.08. Found: C, 49.11; H, 2.93; N, 22.54.

2-Yl-(*p*-chlorobenzylidene hydrazino)-3(H)-4-(methoxy carbonyl methylene)-imidazol-5-one (2g).

p-chlorobenzaldehyde guanylhydrazone and DMAD, this compound was obtained as yellow powder, Yield 92%, mp 194-196 ^oC; IR: NH 3421, 3391, CO 1697 cm^{-1}; ^1H nmr (300 MHz, DMSO-d_6) δ 9.20 (br, 1H, NH), 8.53 (br, 1H, NH), 8.13 (s, 1H, N=CH), 7.91 (d, 2H, *J* = 8.0 Hz, Ar-H), 7.55 (d, 2H, *J* = 8.0Hz, Ar-H), 3.53 (s, 3H, OMe); ^{13}C nmr (300 MHz, DMSO-d_6) δ 49.77, 72.16, 94.26, 126.79, 128.14, 129.61, 134.10, 134.20, 149.56, 157.50, 162.56, 165.47, 171.55; MS: m/z 306 (M^+); Anal. Calcd for $C_{13}H_{11}N_4O_3Cl$: C, 50.98; H, 3.59; N, 18.30. Found: C, 50.92; H, 3.64; N, 18.42.

RESULTS AND DISCUSSION

Initially,we first carried out the reaction of DMAD with thiosemicarbazone of arylaldehydes in methanol to obtain a single compound in each case which was characterized to be, 1-(arylideneamino)-5-(methoxy carbonyl methylene)-2-thione-imidazol-4-one **(1a-h)** in high yield (Scheme 1). The structure of **(1)** was determined by ^1H nmr, ir, mass spectra and elemental analyses. The ir spectra of **(1a-h)** exhibited SH absorptions (2760-2779 cm^{-1})، ^1H nmr spectra exhibited exo methylen protons (s, δ 6.59-6.92 ppm) and NH (br, δ 11.01-13.19 ppm). All of mass spectra exhibited M^+. Elemental analysis supported the assigned structures.

We have also found that the reaction of guanylhydrazone derivatives of arylaldehydes with DMAD in methanol afford 2-yl (aryliden hydrazino)-3(H)-4-(methoxy carbonyl methylene)- imidazol-5-ones **(2a-g)** in good yields (Scheme 2). The structure of **(2)** was confirmed by ^1H nmr , ir, mass spectra and elemental analysis.

CONCLUSION

The method described in this paper,allows the prepartion of unique substituted imidazole from commerical and avialable DMAD and easy to prepare thiosemicarbazone and

guanylhydrazone of aldehydes.The important aspects of this protocol ,are high yielding, mild reaction conditions , availability of the percursors and purity of the obtained products with no further crystalization.

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