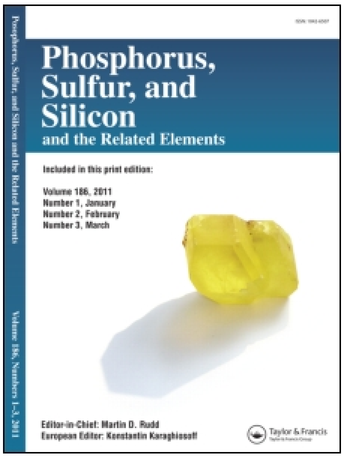

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Synthesis and Fungicidal Activities of 2-Alkylthio-5-phenylmethylene-4H-imidazol-4-ones

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SYNTHESIS AND FUNGICIDAL ACTIVITIES OF 2-ALKYLTHIO-5-PHENYLMETHYLENE-4H-IMIDAZOL-4-ONES

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2-Alkylthio-5-phenylmethylidene-4H-imidazol-4-ones ***4*** *were synthesized by S-alkylation of 2-thioxo-3-aryl-4-imidazolidinones* ***3****, which were obtained via cyclization of isothiocyanate* ***2*** *with aromatic primary amines.* ***3*** *and* ***4*** *exhibited good fungicidal activity against* Physalospora piricola.

Keywords: 4H-Imidazol-4-ones; alkylation; aza-Wittig reaction; fungicidal activities; synthesis

4H-Imidazol-4-ones are important heterocycles having biological and pharmaceutical activities,[1–3] and some 2-alkylthioimidazolones show significant fungicidal activities.[4–6] However, most of the 2-alkylthioimidazolones reported are of the 5,5-disubstituted type and were generally synthesized from corresponding α-amino acetic acid[6,7] (Eq. 1). Unfortunately, 5-arylmethylidene-2-alkylthioimidazolones cannot be prepared by this general method for the corresponding starting material needed would be unstable vinyl amino acetic acids. Recently, we are interested in the synthesis of biologically active imidazolones via tandem aza-Wittig reaction.[8–10] Here we report a new efficient synthesis and fungicidal activity of some new 5-arylmethylidene-2-alkylthioimidazolone derivatives from the stable vinyliminophosphorane **1**.

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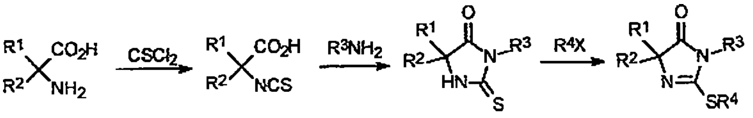
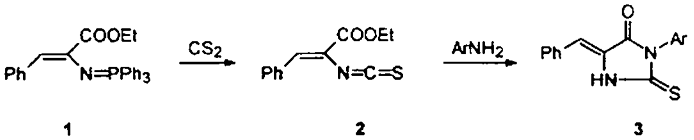


(1)

RESULTS AND DISCUSSION

The easily accessible vinyliminophosphorane **1** reacted with carbon disulfide to give vinyl isothiocyanate **2**. The reaction of **2** with aromatic primary amines took place at refluxing acetonitrile in the presence of potassium carbonate to give 2-thioxo-4-imidazolidinones **3** in 75–85% yields (Table I).



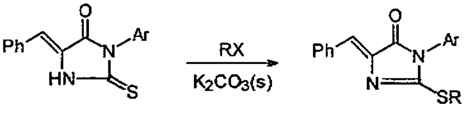
(2)

TABLE I Preparation of 2-Thioxo-4-imidazolidinones **3** and 4H-Imidazol-4-ones **4**

Entry	Ar	RX	Condition	Yield (%)[a]	m.p. (°C)
3a	Ph		80°C/2 h	85	207–208
3b	3-Cl-Ph		80°C/2 h	75	244–245
4a	Ph	*n*-BuBr	60°C/4 h	80	121–122
4b	Ph	*n*-PrBr	50°C/6 h	75	125–126
4c	Ph	*i*-PrBr	50°C/10 h	69	146–147
4d	Ph	*n*-$C_6H_{13}Br$	50°C/8 h	71	71–72
4e	Ph	MeI	r.t./3 h	79	153–155
4f	Ph	EtI	r.t./2 h	70	127–128
4g	Ph	$PhCH_2Cl$	50°C/3 h	90	184–185
4h	Ph	$ClCH_2CN$	50°C/2 h	81	174–175
4i	Ph	$ClCH_2COOEt$	50°C/2 h	85	147–148
4j	Ph	$ClCH_2CONH_2$	50°C/3 h	74	225–227
4k	Ph	$BrCH_2COOMe$	r.t./2 h	75	161–163
4l	Ph	BrCH(Me)COOEt	50°C/2 h	81	111–113
4m	Ph	$BrCH_2COPh$	r.t./2 h	66	202–203
4n	3-Cl-Ph	MeI	r.t./5 h	79	157–159
4o	3-Cl-Ph	$ClCH_2COOEt$	50°C/2 h	77	139–140
4p	3-Cl-Ph	BrCH(Me)COOEt	50°C/2 h	61	91–93

[a] A: Isolated yields of **3a–3b** based on vinyliminophosphorane **1**. B: Purified yields of **4a–4m** based on 2-thioxo-4-imidazolidinone **3a**. C: Purified yields of **4n–4p** based on 2-thioxo-4-imidazolidinone **3b**.

S-Alkylation of **3** with alkyl halides in presence of potassium carbonate provided 2-alkylthio-5-phenylmethylidene-4H-imidazol-4-ones **4** in 61–90% yields. When activated alkylating reagents (RI, $BrCH_2COR$) were used, the alkylation could be carried out at room temperature. When other alkylating reagents were applied, the alkylation had to be carried out at 50–60°C (Table I).

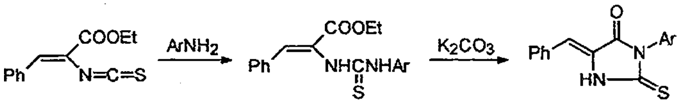


3 **4**

(3)

The structure of **3** and **4** has been characterized spectroscopically. For example, the ^{1}H NMR spectral data in **4e** show the signals of =CH and $-SCH_3$ at 7.00 ppm and 2.67 ppm as single absorption respectively. The chemical shift of aryl hydrogens is 8.16–7.27 ppm with multiple absorption. In the IR spectral data of **4e**, the strong stretching peak of imidazolone C=O appears at 1720 cm^{-1}. The stretching vibration of C=C shows relatively strong absorbtion at about 1633 cm^{-1} due to a resonance effect. The MS of **4e** shows relatively strong molecule ion peak at m/z 295 with 20% abundance.

The formation of **3** can be rationalized in terms of an initial nucleophilic addition of aromatic primary amines to give the intermediate **5** which directly cyclized to give **3** in presence of solid potassium carbonate (Eq. 4).



2 **5** **3**

(4)

The biological activity of **3** and **4** was investigated and the results showed that they exhibited fungicidal activities, especially against *Physalospora piricola*. For example, **4f** and **4m** showed 96% inhibition of *Physalospora piricola* in 50 mg/L (see Table II).

EXPERIMENTAL

Melting points are uncorrected. MS were measured on a HP5988A spectrometer. IR were recorded on a PE-983 infrared spectrometer as KBr pellets with absorption in cm^{-1}. NMR were recorded in $CDCl_3$ on a

TABLE II The Fungicidal Activities of 4-Imidazolidinones **3** and 4H-Imidazol-4-ones **4** (50 mg/L, relative inhibition %)

Entry	*Fusarium oxysporum*	*Gibberella zeae*	*Cercospora beticola sacc*	*Physalospora piricola*	*Pellicularia sasakii*
3a	30	22	25	75	0
3b	20	17	17	50	14
4a	47	62	66	71	94
4b	75	83	61	92	70
4c	44	83	84	92	78
4d	38	62	61	88	74
4e	50	81	84	79	57
4f	59	79	66	96	26
4g	56	62	61	79	35
4h	50	76	61	79	94
4i	38	64	73	92	78
4j	50	64	55	83	13
4k	63	67	84	92	83
4l	34	48	36	83	57
4m	47	98	61	96	35
4n	56	64	86	88	0
4o	63	67	80	92	44
4p	47	76	84	88	70

Varian XL-200 spectrometer and resonances are given in ppm (δ) relative to TMS. Elemental analyses were taken on a Perkin-Elmer 2400 CHN Elementary Analysis Instrument. CS_2 is poisonous and a good hood should be used.

Preparation of Vinyliminophosphorane 1

Vinyliminophosphorane **1** was prepared by the Staudinger reaction of vinyl azide and triphenyl phosphine according to the literature report.[11] m.p. 148–150°C (Lit.[11] m.p. 149°C.)

Preparation of 2-Thioxo-3-aryl-4-imidazolidinones 3

To a solution of vinyliminophosphorane **1**[12] (2.25 g, 5 mmol) in dry methylene chloride (15 mL) was added excess carbon disufide (5 mL). After the reaction mixture was refluxed for 28 h, the solvent was removed under reduced pressure and ether/petroleum ether (1:2, 20 mL) was added to precipitate triphenylphosphine oxide which was removed by filtration. The filtrate was evaporated to give isothiocyanate **2**, which was used directly without further purification. To the solution of **2** in CH_3CN (15 mL) was added $ArNH_2$ (5 mmol) and solid potassium carbonate (0.05 g). The mixture was stirred for 2 h at refluxing temperature and filtered; the filtrate was then evaporated in vacua and the residue was recrystallized from methylene chloride/petroleum ether to give 2-thioxo-3-aryl-4-imidazolidinones **3**.

2-Thioxo-3-pheny-5-phenylmethylene-4-imidazolidinone (3a)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.99 (s, 1H, N—H), 7.49–7.22 (m, 10H, Ar—H), 6.80 (s, 1H, =CH); IR (cm^{-1}), 3217, 1744, 1645; MS (m/z), 280 (M^+, 5%), 136 (17%), 117 (100%). Elemental Anal. Calcd. for $C_{16}H_{12}N_2OS$: C, 68.57; H, 4.28; N, 10.00. Found: C, 68.39; H, 4.17; N, 9.86.

2-Thioxo-3-(3-chloropheny)-5-phenylmethylene-4-imidazolidinone (3b)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.84 (s, 1H, N—H), 7.60–7.22 (m, 9H, Ar—H), 6.81 (s, 1H, =CH); IR (cm^{-1}), 3223, 1745, 1642; MS (m/z), 314 (M^+, 3%), 168 (12%), 117 (53%), 77 (99%), 45 (100%). Elemental Anal. Calcd. for $C_{16}H_{11}ClN_2OS$: C, 61.05; H, 3.50; N, 8.90. Found: C, 59.97; H, 3.39; N, 9.08.

Preparation of 2-Alkylthio-4H-imidazol-4-ones 4

A mixture of **3** (4 mmol), alkyl halide (5 mmol) and solid potassium carbonate (1.11 g, 8 mmol) in CH_3CN (30 mL) was stirred for 2–10 h at room temperature or 50–60°C and filtered, the filtrate was condensed and the residue was recrystallized from methylene chloride/petroleum ether to give 2-alkylthio-4H-imidazol-4-ones **4**.

2-(n-Butylthio)-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4a)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.17–7.22 (m, 10H, Ar—H), 6.99 (s, 1H, =CH), 3.31 (t, 2H, SCH_2, $J = 7.3$ Hz), 1.86–1.44 (m, 4H, CH_2CH_2), 0.99 (t, 3H, CH_3, $J = 7.3$ Hz); IR (cm^{-1}), 1728, 1635; MS (m/z), 337 ($M^+ + 1$, 2%), 280 (10%), 136 (37%), 117 (100%). Elemental Anal. Calcd. for $C_{20}H_{20}N_2OS$: C, 71.40; H, 5.99; N, 8.33. Found: C, 71.36; H, 5.73; N, 8.48.

2-(n-Propylthio)-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4b)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.16–7.21 (m, 10H, Ar—H), 6.99 (s, 1H, =CH), 3.29 (t, 2H, SCH_2, $J = 7.3$ Hz), 1.92–1.81 (m, 2H, CH_2), 1.08 (t, 3H, CH_3, $J = 7.3$ Hz); IR (cm^{-1}), 1723, 1635; MS (m/z), 323 ($M^+ + 1$, 3%), 280 (10%), 136 (51%), 117 (100%). Elemental Anal.

Calcd. for $C_{19}H_{18}N_2OS$: C, 70.78; H, 5.63; N, 8.69. Found: C, 70.84; H, 5.72; N, 8.57.

2-(i-Propylthio)-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4c)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.15–7.20 (m, 10H, Ar—H), 6.99 (s, 1H, =CH), 4.21–4.14 (m, 1H, SCH, J = 6.8 Hz), 1.50 (d, 6H, $2CH_3$, J = 6.8 Hz); IR (cm^{-1}), 1721, 1632; MS (m/z), 323 (M^+ + 1, 9%), 280 (19%), 136 (43%), 117 (100%). Elemental Anal. Calcd. for $C_{19}H_{18}N_2OS$: C, 70.78; H, 5.63; N, 8.69. Found: C, 70.60; H, 5.85; N, 8.74.

2-(n-Hexylthio)-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4d)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.16–7.21 (m, 10H, Ar—H), 6.99 (s, 1H, =CH), 3.30 (t, 2H, SCH_2, J = 7.3 Hz), 1.91–0.87 (m, 11H, $(CH_2)_4CH_3$); IR (cm^{-1}), 1718, 1636; MS (m/z), 365 (M^+ + 1, 2%), 318 (4%), 281 (18%), 136 (18%), 117 (66%), 32 (100%). Elemental Anal. Calcd. for $C_{22}H_{24}N_2OS$: C, 72.49; H, 6.64; N, 7.69. Found: C, 72.45; H, 6.77; N, 7.62.

2-Methylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4e)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.16–7.27 (m, 10H, Ar—H), 7.00 (s, 1H, =CH), 2.67 (s, 3H, SCH_3); IR (cm^{-1}), 1720, 1633; MS (m/z), 295 (M^+ + 1, 20%), 248 (6%), 150 (72%), 135 (54%), 116 (34%), 77 (100%). Elemental Anal. Calcd. for $C_{17}H_{14}N_2OS$: C, 69.36; H, 4.79; N, 9.52. Found: C, 69.15; H, 4.69; N, 9.63.

2-Ethylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4f)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.16–7.27 (m, 10H, Ar—H), 7.00 (s, 1H, =CH), 3.32 (q, 2H, SCH_2, J = 7.3 Hz), 1.47 (t, 3H, CH_3, J = 7.3 Hz); IR (cm^{-1}), 1721, 1630; MS (m/z), 309 (M^+ + 1, 10%), 280 (7%), 248 (3%), 204 (7%), 136 (53%), 117 (50%), 32 (100%). Elemental Anal. Calcd. for $C_{18}H_{16}N_2OS$: C, 70.10; H, 5.23; N, 9.08. Found: C, 70.32; H, 5.18; N, 9.26.

2-Benzylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4g)

Yellow crystals, ^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.19–7.19 (m, 15H, Ar—H), 7.03 (s, 1H, =CH), 4.55 (s, 2H, SCH_2Ph); IR (cm^{-1}), 1730, 1634; MS (m/z), 371 (M^+ + 1, 1%), 167 (3%), 116 (8%), 77 (61%), 32 (100%).

Elemental Anal. Calcd. for $C_{23}H_{18}N_2OS$: C, 74.57; H, 4.90; N, 7.56. Found: C, 74.63; H, 4.78; N, 7.74.

2-Cyanomethylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4h)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.15–7.21 (m, 10H, Ar—H), 7.11 (s, 1H, =CH), 4.04 (s, 2H, SCH_2CN); IR (cm^{-1}), 2258, 1726, 1627; MS (m/z), 320 ($M^+ + 1$, 10%), 280 (4%), 175 (15%), 144 (24%), 135 (100%), 116 (77%). Elemental Anal. Calcd. for $C_{18}H_{13}N_3OS$: C, 67.69; H, 4.10; N, 13.16. Found: C, 67.62; H, 4.32; N, 13.21.

2-Ethoxycarbonylmethylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4i)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.13–7.22 (m, 10H, Ar—H), 7.02 (s, 1H, =CH), 4.24 (q, 2H, $COOCH_2$, J = 7.3 Hz), 4.05 (s, 2H, SCH_2COO), 1.30 (t, 3H, CH_3, J = 7.3 Hz); IR (cm^{-1}), 1737, 1722, 1634; MS (m/z), 367 ($M^+ + 1$, 9%), 294 (14%), 248 (5%), 135 (37%), 116 (92%), 77 (100%). Elemental Anal. Calcd. for $C_{20}H_{18}N_2O_3S$: C, 65.56; H, 4.95; N, 7.64. Found: C, 65.43; H, 4.74; N, 7.83.

2-Aminocarbonylmethylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4j)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.08–7.22 (m, 10H, Ar—H), 7.08 (s, 1H, =CH), 5.51 (s, 2H, NH_2), 3.91 (s, 2H, SCH_2CO); IR (cm^{-1}), 3305, 3261, 1709, 1678, 1630; MS (m/z), 338 ($M^+ + 1$, 2%), 280 (2%), 193 (2%), 135 (7%), 116 (26%), 77 (32%), 32 (100%). Elemental Anal. Calcd. for $C_{18}H_{15}N_3O_2S$: C, 64.08; H, 4.48; N, 12.45. Found: C, 64.30; H, 4.37; N, 12.63.

2-Methoxycarbonylmethylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4k)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.13–7.22 (m, 10H, Ar—H), 7.02 (s, 1H, =CH), 4.04 (s, 2H, SCH_2COO), 3.79 (s, 3H, CH_3); IR (cm^{-1}), 1737, 1724, 1637; MS (m/z), 353 ($M^+ + 1$, 12%), 294 (14%), 248 (4%), 208 (8%), 135 (44%), 116 (75%), 32 (100%). Elemental Anal. Calcd. for $C_{19}H_{16}N_2O_3S$: C, 64.76; H, 4.58; N, 7.95. Found: C, 64.63; H, 4.56; N, 8.12.

2-Ethoxycarbonyl(methyl)methylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4l)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.13–7.21 (m, 10H, Ar—H), 7.00 (s, 1H, =CH), 4.65 (q, 1H, SCHCOO, J = 7.7 Hz), 4.24–4.18

(q, 2H, $COOCH_2$, J = 7.7 Hz), 1.69 (d, 3H, $CH\underline{CH_3}$, J = 7.7 Hz), 1.24 (t, 3H, $OCH_2\underline{CH_3}$, J = 7.7 Hz); IR (cm^{-1}), 1739, 1723, 1635; MS (m/z), 381 (M^+ + 1, 1%), 308 (2%), 280 (2%), 136 (6%), 116 (18%), 44 (100%). Elemental Anal. Calcd. for $C_{21}H_{20}N_2O_3S$: C, 66.30; H, 5.30; N, 7.36. Found: C, 66.17; H, 5.58; N, 7.19.

2-Benzoylmethylthio-3-pheny-5-phenylmethylene-4H-imidazol-4-one (4m)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.09–7.03 (m, 15H, Ar—H), 6.96 (s, 1H, =CH), 4.77 (s, 2H, SCH_2COPh); IR (cm^{-1}), 1721, 1694, 1634; MS (m/z), 399 (M^+ + 1, 1%), 294 (3%), 135 (6%), 116 (12%), 105 (71%), 32 (100%). Elemental Anal. Calcd. for $C_{24}H_{18}N_2O_2S$: C, 72.34; H, 4.55; N, 7.03. Found: C, 72.38; H, 4.37; N, 7.26.

2-Methylthio-3-(3-chloropheny)-5-phenylmethylene-4H-imidazol-4-one (4n)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.15–7.21 (m, 9H, Ar—H), 7.00 (s, 1H, =CH), 2.70 (s, 3H, SCH_3); IR (cm^{-1}), 1716, 1637; MS (m/z), 329 (M^+ + 1, 2%), 184 (4%), 170 (4%), 116 (6%), 44 (100%). Elemental Anal. Calcd. for $C_{17}H_{13}ClN_2OS$: C, 62.10; H, 3.98; N, 8.52. Found: C, 62.26; H, 3.74; N, 8.64.

2-Ethoxycarbonylmethylthio-3-(3-chloropheny)-5-phenylmethylene-4H-imidazol-4-one (4o)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.12–7.19 (m, 9H, Ar—H), 7.02 (s, 1H, =CH), 4.24 (q, 2H, $COOCH_2$, J = 7.3Hz), 4.05 (s, 2H, SCH_2COO), 1.30 (t, 3H, CH_3, J = 7.3 Hz); IR (cm^{-1}), 1730, 1718, 1636; MS (m/z), 401 (M^+ + 1, 1%), 328 (1%), 154 (1%), 116 (5%), 31 (100%). Elemental Anal. Calcd. for $C_{20}H_{17}ClN_2O_3S$: C, 59.92; H, 4.27; N, 6.99. Found: C, 59.73; H, 4.17; N, 7.21.

2-Ethoxycarbonyl(methyl)methylthio-3-(3-chloropheny)-5-phenylmethylene-4H-imidazol-4-one (4p)

Yellow crystals, ^1H NMR ($CDCl_3$, 200 MHz) δ 8.12–7.21 (m, 9H, Ar—H), 7.01 (s, 1H, =CH), 4.65 (q, 1H, SCHCOO, J = 7.7 Hz), 4.28–4.13 (q, 2H, $COOCH_2$, J = 7.3 Hz), 1.70 (d, 3H, $CH\underline{CH_3}$, J = 7.7 Hz), 1.28 (t, 3H, $OCH_2\underline{CH_3}$, J = 7.3 Hz); IR (cm^{-1}), 1742, 1716, 1636; MS (m/z), 415 (M^+ + 1, 7%), 342 (15%), 314 (10%), 250 (5%), 144 (25%), 116 (100%). Elemental Anal. Calcd. for $C_{21}H_{19}ClN_2O_3S$: C, 60.79; H, 4.62; N, 6.75. Found: C, 60.53; H, 4.83; N, 6.67.

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