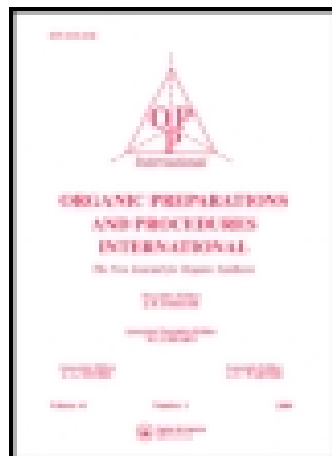




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NEW EFFICIENT SYNTHESIS OF 2-ALKYLTHIO-5-BENZYLIDENE-4H-IMIDAZOLIN-4-ONES

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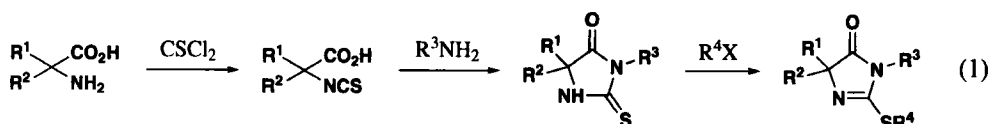
NEW EFFICIENT SYNTHESIS OF 2-ALKYLTHIO-5-BENZYLIDENE-4H-IMIDAZOLIN-4-ONES

Submitted by
(10/25/02)

Ming-Wu Ding*, Yong Sun, Xiao-Peng Liu, Zhao-Jie Liu

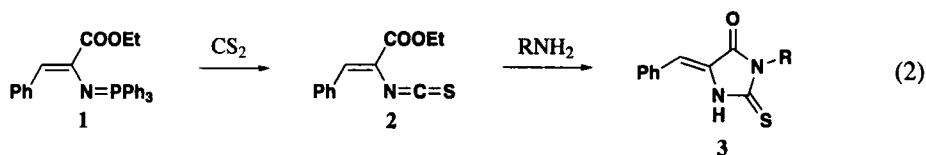
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4H-Imidazolin-4-ones are important heterocycles having biological and pharmaceutical activities,¹⁻⁶ and some 2-alkylthioimidazolinones show significant fungicidal activities.⁷⁻⁹ However, most of the 2-alkylthioimidazolinones reported are of the 5,5-disubstituted type and were generally synthesized from corresponding α -aminoacetic acid^{9,10} (Eq. 1). Unfortunately, 5-benzylidene-2-alkylthioimidazolinones cannot be prepared by this general method for the



corresponding starting material would be unstable vinyl amino acetic acids. Recently, we became interested in the synthesis of biologically active imidazolinones *via* tandem aza-Wittig reaction¹¹⁻¹³ and now report a new efficient preparation of 5-benzylidene-2-alkylthioimidazolinones from the stable vinyliminophosphorane 1.

The easily accessible vinyliminophosphorane 1¹⁴ reacted with carbon disulfide to give vinyl isothiocyanate 2. While the reaction of 2 with aliphatic primary amines took place smoothly at room temperature to give 2-thioxo-4-imidazolidinones 3 in 65-95% yields, aromatic primary amines required reflux in acetonitrile in the presence of potassium carbonate (Eq. 2).



The results are listed in Table 1. Some of 2-thioxo-4-imidazolidinones 3 were also compared with authentic samples prepared previously by condensation of 2-thiohydantoin with aromatic aldehyde,¹⁵ or by reaction of 4-benzylidene-2-benzylthio-2-thiazolin-5-ones with various primary aromatic amines in the presence of sodium acetate in acetic acid.¹⁶

S-Alkylation of 3d with alkyl halides in the presence of potassium carbonate provided 2-alkylthio-5-benzylidene-4H-imidazolin-4-ones 4 in 63-87% yields. While activated alkylating agents (RI, BrCH₂COR) reacted at room temperature, the other alkylating reagents required heating at 50-60°C (Table 2).

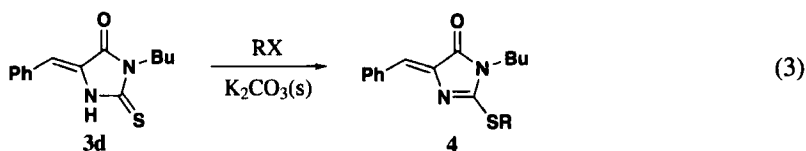


Table 1. Preparation of 3-Substituted 2-Thioxo-4-imidazolidinones (3) from Vinyl Isothiocyanate (2)

Compounds	R	Condition	Yield (%) ^a	mp. (lit.) (°C)
3a	Et	r.t./30 min	65	167-168
3b	<i>n</i> -Pr	r.t./20 min	95	120-121
3c	<i>i</i> -Pr	r.t./30 min	88	122-124
3d	<i>n</i> -Bu	r.t./50 min	72	107-108
3e	Cyclohexyl	r.t./10 min	95	160-162 (162-164 ¹⁵)
3f	4-MeC ₆ H ₄	80°C/2 hr	61	187-188 (185 ¹⁶)
3g	2-MeC ₆ H ₄	80°C /2 hr	64	165-166 (164 ¹⁶)
3h	4-ClC ₆ H ₄	80°C /2 hr	95	259-261 (257 ¹⁶)
3i	4-BrC ₆ H ₄	80°C /3 hr	85	245-246 (244 ¹⁶)

a) Yields based on vinyliminophosphorane 1.

Table 2. Alkylation of 3d to 2-Alkylthio-4H-imidazolin-4-ones (4)

Compounds	RX	Condition	Yield (%) ^a	mp (°C)
4a	MeI	r.t./2 hr	70	113-114
4b	<i>n</i> -C ₆ H ₁₃ Br	60°C /3 hr	63	35-36
4c	PhCH ₂ Cl	50°C /4 hr	77	82-83
4d	ClCH ₂ CN	50°C /2 hr	67	98-100
4e	BrCH ₂ COPh	r.t./1 hr	76	161-162
4f	ClCH ₂ CONH ₂	50°C /4 hr	64	163-165
4g	BrCH ₂ COOMe	r.t./2 hr	87	82-83
4h	ClCH ₂ COOEt	50°C /2 hr	78	84-85
4i	BrCH(Me)COOEt	50°C /2 hr	82	75-76

a) Yields based on 2-thioxo-4-imidazolidinone 3d.

EXPERIMENTAL SECTION

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 CDCl₃ on a Varian XL-200 spectrometer and resonances are given in ppm (δ) relative to TMS. Elemental analyses were taken on a Perkin-Elmer 2400 CHN Elemental Analysis Instrument. CS₂ is poisonous and an efficient hood should be used.

Preparation of 3-Alkyl-2-thioxo-4-imidazolidinones (3a-e). General Procedure.- To a solution of vinyliminophosphorane **1**¹⁴ (2.25 g, 5 mmol) in dry methylene chloride (15 mL) was added excess carbon disulfide (5 mL). After the reaction mixture was refluxed for 28 hours, the solvent was removed under reduced pressure and ether/petroleum ether (1:2, 20 mL) was added to precipitate triphenylphosphine sulfide which was removed by filtration. The filtrate was evaporated to give isothiocyanate **2**, which was used directly without further purification. To a solution of the crude **2** in CH₃CN (15 mL) was added RNH₂ (5 mmol) and the mixture was allowed to stand for 10~50 min at room temperature. The precipitated solid was collected and washed with water and ethanol, crystallized from methylene chloride/petroleum ether to give 3-alkyl-2-thioxo-4-imidazolidinone **3a-e**.

Compound **3a**: yield 65%, yellow crystals, mp. 167-168°C. ¹H NMR (CDCl₃, 200 MHz): δ 8.82 (s, 1H, N-H), 7.41~7.22 (m, 5H, Ar-H), 6.70 (s, 1H, =CH), 3.95 (q, 2H, J = 7.8Hz, NCH₂), 1.29 (t, 3H, J = 7.8Hz, CH₃). IR (KBr, cm⁻¹): 3255 (N-H), 1708 (C=O), 1648 (C=C). MS (m/z, %): 232 (M⁺, 93), 203 (18), 189 (7), 117 (100).

Anal. Calcd for C₁₂H₁₂N₂OS: C, 62.05; H, 5.21; N, 12.06. Found: C, 62.13; H, 5.39; N, 11.94

Compound **3b**: yield 95%, yellow crystals, mp. 120-121°C, ¹H NMR (CDCl₃, 200 MHz): δ 8.82 (s, 1H, N-H), 7.48~7.24 (m, 5H, Ar-H), 6.67 (s, 1H, =CH), 3.84 (t, 2H, J = 7.3Hz, NCH₂), 1.75~0.89 (m, 5H, CH₂CH₃). IR (KBr, cm⁻¹): 3252 (N-H), 1706 (C=O), 1646 (C=C). MS (m/z, %): 246 (M⁺, 100), 231 (10), 213 (88), 203 (97), 160 (72), 117 (92).

Anal. Calcd for C₁₃H₁₄N₂OS: C, 63.39; H, 5.73; N, 11.37. Found: C, 63.28; H, 5.79; N, 11.18

Compound **3c**: yield 88%, yellow crystals, mp. 122-124°C, ¹H NMR (CDCl₃, 200 MHz): δ 8.81 (s, 1H, N-H), 7.47~7.24 (m, 5H, Ar-H), 6.60 (s, 1H, =CH), 4.99~4.91 (m, 1H, NCH), 1.50 (d, 6H, J = 6.8Hz, CH₃). IR (KBr, cm⁻¹): 3257 (N-H), 1709 (C=O), 1649 (C=C). MS (m/z, %): 246 (M⁺, 64), 213 (5), 203 (58), 160 (8), 117 (100).

Anal. Calcd for C₁₃H₁₄N₂OS: C, 63.39; H, 5.73; N, 11.37. Found: C, 63.21; H, 5.64; N, 11.44

Compound **3d**: yield 72%, yellow crystals, mp. 107-108°C, ¹H NMR (CDCl₃, 200 MHz): δ 8.82 (s, 1H, N-H), 7.41~7.23 (m, 5H, Ar-H), 6.68 (s, 1H, =CH), 3.88 (t, 2H, J = 7.7Hz, NCH₂), 1.77~0.84 (m, 7H, CH₂CH₂CH₃). IR (KBr, cm⁻¹): 3248 (N-H), 1707 (C=O), 1647 (C=C). MS (m/z, %): 260 (M⁺, 35), 227 (100), 203 (14), 160 (12), 117 (64).

Anal. Calcd for C₁₄H₁₆N₂OS: C, 64.59; H, 6.19; N, 10.76. Found: C, 64.51; H, 6.28; N, 10.56

Compound **3e**: yield 95%, yellow crystals, mp. 160-162°C, (*lit.*¹⁵ 162-164°C), ¹H NMR (CDCl₃, 200 MHz): δ 8.80 (s, 1H, N-H), 7.45~7.23 (m, 5H, Ar-H), 6.60 (s, 1H, =CH), 4.59~4.47 (m, 1H, NCH), 2.38~0.94 (m, 10H, (CH₂)₅). IR (KBr, cm⁻¹): 3256 (N-H), 1709 (C=O), 1648 (C=C). MS (m/z, %): 286 (M⁺, 98), 253 (13), 203 (99), 160 (46), 117 (100).

Anal. Calcd for C₁₆H₁₈N₂OS: C, 67.10; H, 6.33; N, 9.78. Found: C, 67.24; H, 6.37; N, 9.64

Preparation of 3-Aryl-2-thioxo-4-imidazolidinones (3f-i). General Procedure.- To the solution of **2** in CH₃CN (15 mL) was added ArNH₂ (5 mmol) and solid potassium carbonate (0.05 g).

The mixture was stirred for 2–3 hr at reflux and filtered; the filtrate was then evaporated *in vacuo* and the residue was recrystallized from methylene chloride/petroleum ether to give 3-aryl-2-thioxo-4-imidazolidinone **3f-i**.

Compound **3f**: yield 61%, yellow crystals, mp. 187–188°C, (*lit.*¹⁶ 185°C), ¹H NMR (CDCl₃, 200 MHz): δ 9.19 (s, 1H, N-H), 7.56–7.16 (m, 9H, Ar-H), 6.78 (s, 1H, =CH), 2.39 (s, 3H, CH₃). IR (KBr, cm⁻¹): 3231 (N-H), 1746 (C=O), 1642 (C=C). MS (m/z, %): 294 (M⁺, 100), 265 (38), 236 (14), 150 (48), 132 (50), 116 (99).

Anal. Calcd for C₁₇H₁₄N₂OS: C, 69.36; H, 4.79; N, 9.52. Found: C, 69.27; H, 4.84; N, 9.58

Compound **3g**: yield 64%, yellow crystals, mp. 165–166°C, (*lit.*¹⁶ 164°C), ¹H NMR (CDCl₃, 200 MHz): δ 9.30 (s, 1H, N-H), 7.46–7.10 (m, 9H, Ar-H), 6.78 (s, 1H, =CH), 2.16 (s, 3H, CH₃). IR (KBr, cm⁻¹): 3234 (N-H), 1748 (C=O), 1645 (C=C). MS (m/z, %): 294 (M⁺, 92), 279 (39), 261 (100), 233 (78), 148 (50), 116 (91).

Anal. Calcd for C₁₇H₁₄N₂OS: C, 69.36; H, 4.79; N, 9.52. Found: C, 69.45; H, 4.82; N, 9.35

Compound **3h**: yield 95%, yellow crystals, mp. 259–261°C, (*lit.*¹⁶ 257°C), ¹H NMR (CDCl₃, 200 MHz): δ 8.74 (s, 1H, N-H), 7.53–7.17 (m, 9H, Ar-H), 6.80 (s, 1H, =CH). IR (KBr, cm⁻¹): 3220 (N-H), 1744 (C=O), 1641 (C=C). MS (m/z, %): 314 (M⁺, 80), 316 (28), 285 (5), 170 (20), 125 (27), 117 (100).

Anal. Calcd for C₁₆H₁₁ClN₂OS: C, 61.05; H, 3.52; N, 8.90. Found: C, 61.18; H, 3.43; N, 8.93

Compound **3i**: yield 85%, yellow crystals, mp. 245–246°C, (*lit.*¹⁶ 244°C), ¹H NMR (CDCl₃, 200 MHz): δ 8.90 (s, 1H, N-H), 7.77–7.08 (m, 9H, Ar-H), 6.81 (s, 1H, =CH). IR (KBr, cm⁻¹): 3221 (N-H), 1741 (C=O), 1640 (C=C). MS (m/z, %): 360 (M⁺, 70), 358 (75), 214 (12), 160 (22), 135 (25), 125 (39), 117 (100).

Anal. Calcd for C₁₆H₁₁BrN₂OS: C, 53.50; H, 3.09; N, 7.80. Found: C, 53.36; H, 3.02; N, 7.97

Preparation of 2-Alkylthio-4H-imidazolin-4-ones 4. General Procedure.— A mixture of **3d** (1.04 g, 4 mmol), alkyl halide (5 mmol) and solid potassium carbonate (1.11 g, 8 mmol) in CH₃CN (30 mL) was stirred for 1–4 hr 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-imidazolin-4-ones **4**.

Compound **4a**: yield 70%, yellow crystals, mp. 113–114°C, ¹H NMR (CDCl₃, 200 MHz): δ 8.12–7.32 (m, 5H, Ar-H), 6.89 (s, 1H, =CH), 3.56 (t, 2H, J = 6.8 Hz, NCH₂), 2.72 (s, 3H, SCH₃), 1.67–0.90 (m, 7H, CH₂CH₂CH₃). IR (KBr, cm⁻¹): 1712 (C=O), 1636 (C=C). MS (m/z, %): 274 (M⁺, 92), 259 (70), 245 (68), 227 (90), 185 (64), 116 (96), 74 (100).

Anal. Calcd for C₁₅H₁₈N₂OS: C, 65.66; H, 6.61; N, 10.21. Found: C, 65.57; H, 6.79; N, 10.24

Compound **4b**: yield 63%, yellow crystals, mp. 35–36°C, ¹H NMR (CDCl₃, 200 MHz): δ 8.12–7.32 (m, 5H, Ar-H), 6.88 (s, 1H, =CH), 3.56 (t, 2H, J = 7.3 Hz, NCH₂), 3.32 (t, 2H, J = 7.3 Hz, SCH₂), 1.92–0.98 (m, 18H, NCH₂CH₂CH₂CH₃ and SCH₂(CH₂)₄CH₃). IR (KBr, cm⁻¹): 1712 (C=O), 1635 (C=C). MS (m/z, %): 344 (M⁺, 13), 297 (18), 260 (14), 227 (100), 203 (12), 160 (13).

Anal. Calcd for $C_{20}H_{28}N_2OS$: C, 69.73; H, 8.19; N, 8.13. Found: C, 69.69; H, 8.12; N, 8.27

Compound **4c**: yield 77%, light yellow crystals, mp. 82–83°C, 1H NMR ($CDCl_3$, 200 MHz): δ 8.14–7.22 (m, 10H, Ar-H), 6.93 (s, 1H, =CH), 4.59 (s, 2H, SCH_2Ph), 3.55 (t, 2H, $J = 7.3$ Hz, NCH_2), 1.66–0.89 (m, 7H, $NCH_2CH_2CH_2CH_3$). IR (KBr, cm^{-1}): 1713 (C=O), 1637 (C=C). MS (m/z , %): 350 (M^+ , 41), 317 (33), 259 (15), 227 (56), 160 (10), 144 (16), 91 (100).

Anal. Calcd for $C_{21}H_{22}N_2OS$: C, 71.97; H, 6.33; N, 7.99. Found: C, 71.85; H, 6.22; N, 8.04

Compound **4d**: yield 67%, light yellow crystals, mp. 98–100°C, 1H NMR ($CDCl_3$, 200 MHz): δ 8.10–7.22 (m, 5H, Ar-H), 7.01 (s, 1H, =CH), 4.12 (s, 2H, SCH_2CN), 3.56 (t, 2H, $J = 6.8$ Hz, NCH_2), 1.67–0.92 (m, 7H, $CH_2CH_2CH_3$). IR (KBr, cm^{-1}): 2256 (CN), 1714 (C=O), 1636 (C=C). MS (m/z , %): 299 (M^+ , 19), 260 (18), 227 (23), 160 (20), 144 (62), 116 (100).

Anal. Calcd for $C_{16}H_{17}N_3OS$: C, 64.19; H, 5.72; N, 14.04. Found: C, 64.34; H, 5.79; N, 13.96

Compound **4e**: yield 76%, light yellow crystals, mp. 161–162°C, 1H NMR ($CDCl_3$, 200 MHz): δ 8.09–7.02 (m, 10H, Ar-H), 6.85 (s, 1H, =CH), 4.80 (s, 2H, SCH_2), 3.62 (t, 2H, $J = 7.3$ Hz, NCH_2), 1.73–0.93 (m, 7H, $CH_2CH_2CH_3$). IR (KBr, cm^{-1}): 1714, 1695 (C=O), 1636 (C=C). MS (m/z , %): 378 (M^+ , 11), 273 (21), 227 (16), 134 (16), 116 (19), 105 (100).

Anal. Calcd for $C_{22}H_{22}N_2O_2S$: C, 69.82; H, 5.86; N, 7.40. Found: C, 69.63; H, 5.78; N, 7.47

Compound **4f**: yield 64%, yellow crystals, mp. 163–165°C, 1H NMR ($CDCl_3$, 200 MHz): δ 8.02–7.23 (m, 5H, Ar-H), 6.96 (s, 1H, =CH), 5.82 (s, 2H, NH_2), 3.93 (s, 2H, SCH_2), 3.57 (t, 2H, $J = 7.2$ Hz, NCH_2), 1.67–0.91 (m, 7H, $CH_2CH_2CH_3$). IR (KBr, cm^{-1}): 3310, 3266 (NH_2), 1714, 1675 (C=O), 1637 (C=C). MS (m/z , %): 317 (M^+ , 38), 273 (14), 259 (72), 160 (20), 116 (100).

Anal. Calcd for $C_{16}H_{19}N_3O_2S$: C, 60.55; H, 6.03; N, 13.24. Found: C, 60.51; H, 6.21; N, 13.17

Compound **4g**: yield 87%, light yellow crystals, mp. 82–83°C, 1H NMR ($CDCl_3$, 200 MHz): δ 8.08–7.22 (m, 5H, Ar-H), 6.91 (s, 1H, =CH), 4.07 (s, 2H, SCH_2), 3.79 (s, 3H, OCH_3), 3.59 (t, 2H, $J = 7.2$ Hz, NCH_2), 1.65–0.92 (m, 7H, $CH_2CH_2CH_3$). IR (KBr, cm^{-1}): 1736, 1713 (C=O), 1636 (C=C). MS (m/z , %): 332 (M^+ , 100), 303 (51), 259 (88), 227 (94), 188 (73), 116 (83).

Anal. Calcd for $C_{17}H_{20}N_2O_3S$: C, 61.43; H, 6.06; N, 8.43. Found: C, 61.25; H, 6.11; N, 8.38

Compound **4h**: yield 78%, light yellow crystals, mp. 84–85°C, 1H NMR ($CDCl_3$, 200 MHz): δ 8.08–7.32 (m, 5H, Ar-H), 6.90 (s, 1H, =CH), 4.23 (q, 2H, $J = 6.8$ Hz, OCH_2), 4.08 (s, 2H, SCH_2), 3.58 (t, 2H, $J = 7.3$ Hz, NCH_2), 1.70–0.92 (m, 10H, $NCH_2CH_2CH_2CH_3$ and $COOCH_2CH_3$). IR (KBr, cm^{-1}): 1739, 1713 (C=O), 1633 (C=C). MS (m/z , %): 346 (M^+ , 26), 259 (34), 227 (35), 144 (27), 116 (62), 31 (100).

Anal. Calcd for $C_{18}H_{22}N_2O_3S$: C, 62.41; H, 6.40; N, 8.09. Found: C, 62.32; H, 6.54; N, 8.01

Compound **4i**: yield 82%, yellow crystals, mp. 75–76°C, 1H NMR ($CDCl_3$, 200 MHz): δ 8.08–7.24 (m, 5H, Ar-H), 6.89 (s, 1H, =CH), 4.67 (q, 1H, $J = 7.6$ Hz, SCH), 4.23 (q, 2H, $J = 6.5$ Hz, OCH_2), 3.56 (t, 2H, $J = 7.3$ Hz, NCH_2), 1.76–0.91 (m, 13H, $NCH_2CH_2CH_2CH_3$, $SCHCH_3$ and $COOCH_2CH_3$). IR (KBr, cm^{-1}): 1741, 1714 (C=O), 1635 (C=C). MS (m/z , %): 361 (M^+ +1, 26), 260 (22), 227 (34), 144 (49), 116 (100).

Anal. Calcd for $C_{19}H_{24}N_2O_3S$: C, 63.31; H, 6.71; N, 7.77. Found: C, 63.35; H, 6.64; N, 7.53

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