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Synthesis and Fungicidal Activity of Substituted (*E*)-3-Phenyl-1-(pyridin-3-yl)prop-2-en-1-one (*E*,*Z*)-*O*-Alkyl- and *O*-Benzyloximes

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Abstract—Previously unknown substituted (*E*)-3-phenyl-1-(pyridin-3-yl)prop-2-en-1-one (*E*,*Z*)-*O*-alkyl- and *O*-benzyloximes were synthesized by oximation of (*E*)-3-phenyl-1-(pyridin-3-yl)prop-2-en-1-ones (azachalcones), followed by alkylation of the resulting oximes under different conditions. The synthesized compounds showed a good fungicidal activity.

Keywords: 3-phenyl-1-(pyridin3-yl)prop-2-en-1-ones, azachalcone oximes, O-alkylation of oximes, phase-transfer catalysis, *E*,*Z* isomerism of oximes, fungicidal activity.

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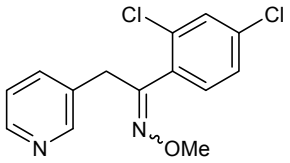
Oxime ethers derived from heterocyclic ketones attract interest due to their potential biological activity. For example, pyrifenox (**1**) has found application as a fungicide with a broad spectrum of action on berry and fruit plants and grape [1], and oxiconasole (**2**) is used for the treatment of superficial mycoses in humans [2]. These compounds inhibit biosynthesis of fungal ergosterol via coordination of the heteroatom to the iron atom in the active site of cytochrome oxidase (CYP51) [1]. Another class of oxime ethers includes fluoxastrobin (**3**) and kresoxim-methyl (**4**) that are widely used to treat crops, vegetables, and fruits [3]. Compounds **3** and **4** bind to cytochrome bc_1 complex thus blocking fungal cell respiration. In recent years, increasing interest is observed to the synthesis of new O-substituted oximes due to their valuable fungicidal properties. 3-Aryl-1-(indol-3-yl)prop-2-en-1-one *O*-alkyloximes [4], 3,3-dimethyl-1-(pyridin-3-yl)butan-2-one *O*-benzoyloximes [5], and 3-phenyl-1-(pyridin-3-yl)propan-1-one *O*-alkyloximes [6] showed fungicidal activity. We previously synthesized (*Z*)-*N*′-methoxypyridine-3-carboximidamides [7, 8] and substituted (pyridin-3-yl)methanone (*E*,*Z*)-*O*-alkyloximes [9], studied their steric structure, and revealed fungicidal activity of these compounds. In continuation of these studies, herein we report the synthesis of a series of new 3-phenyl-1-(pyridin-3-yl)prop-2-en-1-one *O*-alkyl- and *O*-benzyloximes **5a**–**5i** and their fungicidal activity.

Target compounds **5a**–**5i** were synthesized from the corresponding oximes **7a**–**7c** which were prepared according to a conventional procedure, by treatment of azachalcones **6a**–**6c** with hydroxylamine hydrochloride in aqueous ethanol (Scheme 1). Initial azachalcones **6a**–**6c** were obtained in turn by condensation of 3-acetylpyridine with 4-halobenzaldehydes in the presence of 10% sodium hydroxide [10]. The progress of the oximation of **6a**–**6c** and the purity of **7a**–**7c** were monitored by TLC and IR spectroscopy (following the disappearance of the carbonyl stretching vibration band at 1660 cm^{-1}).

Taking into account that alkylating agents with different reactivities were used for the alkylation of **7a**–**7c**, the reactions were performed under different conditions. The benzylation was carried out in a two-phase system (benzene–5% aqueous KOH) using benzyl(triethyl)ammonium chloride as phase-transfer catalyst. However, the alkylation involved not only the oxime functionality but also pyridine nitrogen atom. Furthermore, the reaction was accompanied by hydrolysis of benzyl chloride. As a result, in no case we succeeded in attaining complete conversion of the initial oxime; nevertheless, compounds **5a**–**5c** were isolated in acceptable yields (46.0–69.6%).



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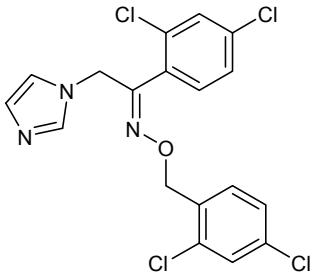

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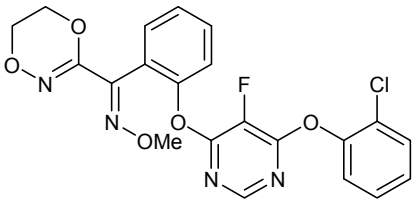

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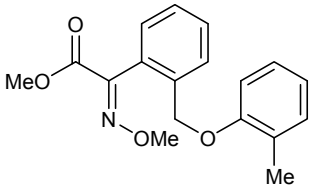

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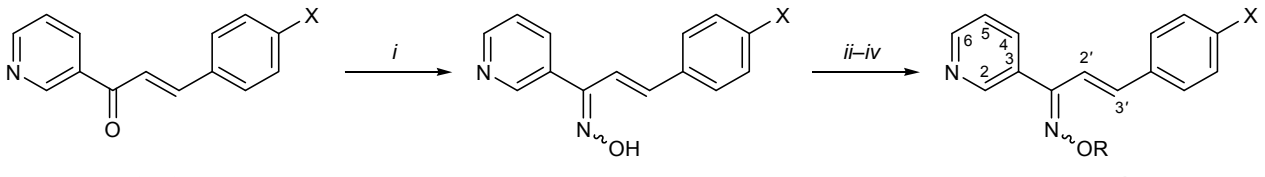
Weaker alkylating agents such as hexyl bromide and cyclohexyl bromide required the use of a stronger base, sodium hydride in DMF. The yields of *O*-cyclohexyloximes **5g**–**5i** (13.6–31.5%) were appreciably lower than the yields of their *O*-hexyl analogs **5d**–**5f** (65.5–74.9%), presumably due to steric hindrances and facile elimination of hydrogen bromide from cyclohexyl bromide.

The unreacted oxime was separated from the alkylation product and solvent by repeated washing with water and 15% aqueous potassium hydroxide. The products were finally purified by dry-column flash chromatography [11] on silica gel (5–40 μm).

Compounds **5a**–**5i** were mixtures of *E* and *Z* isomers at different ratios. The formation of mixtures of isomeric *O*-alkyloximes was also reported in [4]. Signals of the *Z* and *E* isomers were distinguishable in the ^{1}H NMR spectra, and we were able to estimate their ratios by the signal intensities. For this purpose, it was most convenient to use signals of protons at the C=C double bond, which appeared as two pairs of *AB* doublets, as well as signals of 4-H of the pyridine ring (two doublets with spin–spin coupling constants of 7.7–9.7 Hz). The signals were assigned to particular isomers by comparing with the data reported in [4] for 3-aryl-1-(indol-3-yl)prop-2-en-1-one *O*-alkyloximes whose structure was firmly established. Signals of the *E* isomers were located downfield from those of the *Z* isomers. The C=C double bond in all compounds **5a**–**5i** retained its original *E* configuration, as followed from the corresponding ^{1}H–^{1}H coupling constant ($^3J_{trans}$ = 16.1–17.3 Hz).

Compounds **5a**–**5i** were evaluated for their *in vivo* fungicidal activity against five pathogenic fungal cultures belonging to asco-, basidio-, and deuteromycetes: *Venturia inaequalis* (Cooke) Winter, *Rhizoctonia solani* Kühn, *Fusarium oxysporum* Schlecht, *Fusarium moniliforme* Sheldon, and *Helminthosporium sativum* Pammel King et Bakke from the collection of the All-Russian Research Institute of Chemical Means of Plant Protection. Experiments were performed according to a standard procedure [12] using commercial fungicides kresoxim-methyl (**4**) and triadimefon [1-(4-chlorophenoxy)-3,3-dimethyl-1-(1*H*-1,2,4-triazol-1-yl)butan-2-one] as references. It was found that all compounds showed fungicidal activity. The activity of the most

Scheme 1.



6a, **6c** **7a**, **7c** **5a**–**5i**

5, R = $PhCH_2$ (**a**–**c**), C_6H_{13} (**d**–**f**), *cyclo*-C_6H_{11} (**g**–**i**); X = F (**a**, **d**, **g**), Cl (**b**, **e**, **h**), Br (**c**, **f**, **i**); **6**, **7**, X = F (**a**), Cl (**b**), Br (**c**).
Reagents and conditions: *i*: $NH_2OH \cdot HCl$, EtOH, water bath; *ii*: $PhCH_2Cl$, PhH–5% aq. NaOH, BTEAC, reflux; *iii*: $C_6H_{13}Br$, NaH, DMF, 20–60°C; *iv*: *cyclo*-$C_6H_{11}Br$, NaH, DMF, 20–60°C.

Table 1. Radial growth inhibition (% with respect to control) of fungal mycelium *in vivo* in the presence of compounds **5a**–**5i** at a concentration of 30 mg/L

Compound no.	*V. inaequalis*	*R. solani*	*F. oxysporum*	*F. moniliforme*	*H. sativum*
5a	33±5	48±6	22±6	43±3	56±2
5b	28±3	54±5	60±5	90±1	60±2
5c	35±5	–	18±5	30±4	46±3
5d	33±6	3±2	17±5	44±3	37±2
5e	28±2	0	11±2	27±2	28±2
5f	25±3	27±5	16±2	31±5	37±5
5g	28±5	14±4	46±2	49±4	43±5
5h	23±4	59±2	48±2	53±2	46±3
5i	55±1	45±2	45±2	66±2	60±5
4	100	94±1	58±4	57±3	93±2
Triadimefon	60±5	54±4	72±5	85±7	60±5

active derivatives, *O*-benzyloxime **5b** and *O*-cyclohexyloximes **5h** and **5i** was comparable to that of the reference fungicides and in some cases exceeded it.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Bruker AM 300 spectrometer at 300 MHz from solutions in DMSO-d_6. The IR spectra were measured in mineral oil on an IKS-29 spectrometer.

Initial (*E*)-3-phenyl-1-(pyridin-3-yl)prop-2-en-1-ones **6a**–**6c** were synthesized by condensation of 3-acetylpyridine with 4-halobenzaldehydes [10].

[(2*E*)-3-(4-Fluorophenyl)-1-(pyridin-3-yl)prop-2-en-1-ylidene]hydroxylamine (7a). A solution of 0.695 g (10 mmol) of hydroxylamine hydrochloride in 1 mL of water was added to a solution of 1.135 g (5 mmol) of (*E*)-3-(4-fluorophenyl)-1-(pyridin-3-yl)-prop-2-en-1-one (**6a**) in 15 mL of ethanol. The mixture was heated for 1 h on a water bath and cooled to room temperature, a solution of 0.400 g (10 mmol) of sodium hydroxide in 1 mL of water was added, and carbon dioxide was passed through the mixture until pH ~7. The precipitate was filtered off, treated with 5 mL of boiling methanol, 5 mL of tetrahydrofuran was added, the mixture was cooled to room temperature, and the precipitate was filtered off. The filtrate was evaporated to dryness, and the residue was recrystallized from ethanol. Yield 0.566 g (76.8%), mp 130–132°C.

Compounds **7b** and **7c** were synthesized in a similar way.

[(2*E*)-3-(4-Chlorophenyl)-1-(pyridin-3-yl)prop-2-en-1-ylidene]hydroxylamine (7b). Yield 1.106 g (85.6%), mp 147–149°C.

[(2*E*)-3-(4-Bromophenyl)-1-(pyridin-3-yl)prop-2-en-1-ylidene]hydroxylamine (7c). Yield 0.406 g (66.8%), mp 164–166°C.

(1*EZ*,2*E*)-*N*-Benzyloxy-3-(4-fluorophenyl)-1-(pyridin-3-yl)prop-2-en-1-imine (5a). A solution of 0.300 g (2.37 mmol) of benzyl chloride and 0.23 g (1 mmol) of benzyl(triethyl)ammonium chloride (BTEAC) in 10 mL of benzene was added to a solution of 0.484 g (2 mmol) of oxime **7a** in 5% aqueous potassium hydroxide [1.43 g (25.5 mmol) of KOH in 28 mL of water]. The two-phase system was vigorously stirred for 5 h under reflux, the progress of the reaction being monitored by TLC. The mixture was cooled to room temperature, and the organic layer was separated, dried over sodium sulfate, and evaporated. The residue was purified by flash chromatography on silica gel (5–40 μm) using methylene chloride–ethyl acetate (1:1) as eluent. Yield 0.069 g (69.6%), *E*/*Z* ratio 38:62, oily material. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 5.15 s (1.24H, OCH_2, *Z*), 5.32 s (0.76H, OCH_2, *E*), 6.36 d and 7.06 d (1.24H, CH=CH, *Z*, ^3J = 16.5), 6.68 d (0.38H, 3′-H, *E*, *J* = 16.9), 7.27–7.51 m [9.38H, 2′-H (*E*), H_{arom}], 7.56–7.68 m (1H, 5-H), 7.80 d (0.62H, 4-H, Z, *J* = 9.7), 7.91 d (0.38H, 4-H, *E*, *J* = 8.1), 8.55 s (0.62H, 2-H, *Z*), 8.66 d (1H, 6-H, *J* =

4.8), 8.74 s (0.38N, 2-H, *E*). Found, %: C 75.50; H 6.00; N 8.09. $C_{21}H_{17}FN_2O$. Calculated, %: C 75.89; H 5.16; N 8.43.

Compounds **5b** and **5c** were synthesized in a similar way.

(1*EZ*,2*E*)-*N*-Benzyloxy-3-(4-chlorophenyl)-1-(pyridin-3-yl)prop-2-en-1-imine (5b). Yield 0.307 g (59.5%), *E*/*Z* = 45:55, red–brown oil. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 5.15 s (1.10H, OCH_2, *Z*), 5.30 s (0.90H, OCH_2, *E*), 6.35 d and 7.03 d (1.10H, CH=CH, *Z*, *J* = 16.5), 6.67 d and 7.54 d (0.90H, CH=CH, *E*, ^3J = 16.5), 7.27–7.47 m [10.55H, 4-H (*Z*), 5-H, H_{arom}], 7.80 d (0.45H, 4-H, *E*, *J* = 8.1), 8.55 s (0.45H, 2-H, *Z*), 8.66 s (1H, 6-H), 8.74 s (0.55H, 2-H, *E*). Found, %: C 72.01; H 5.11; N 7.75. $C_{21}H_{17}ClN_2O$. Calculated, %: C 72.31; H 4.91; N 8.03.

(1*EZ*,2*E*)-*N*-Benzyloxy-3-(4-bromophenyl)-1-(pyridin-3-yl)prop-2-en-1-imine (5c). Yield 0.362 g (46.0%), *E*/*Z* = 40:60, red–brown oil. ^{1}H NMR spectrum, δ, ppm, (*J*, Hz): 5.16 s (1.20H, OCH_2, *Z*), 5.29 s (0.80H, OCH_2, *E*), 6.33 d and 7.05 d (1.20H, CH=CH, *Z*, ^3J = 16.5), 6.65 d (0.4H, 3′-H, *E*, *J* = 16.1), 7.19–7.50 m [9.60H, 2′-H (*E*), H_{arom}), 7.58–7.72 m (1H, 5-H), 7.81 d (0.60H, 4-H, *Z*, *J* = 9.7), 8.05 d (0.40H, 4-H, *E*, *J* = 8.1), 8.55 s (0.40H, 2-H, *Z*), 8.67 d (1H, 6-H, *J* = 4.4), 8.73 s (0.60H, 2-H, *E*). Found, %: C 64.06; H 4.40; N 7.10. $C_{21}H_{17}BrN_2O$. Calculated, %: C 63.83; H 4.56; N 6.89.

(1*EZ*,2*E*)-3-(4-Fluorophenyl)-*N*-hexyloxy-1-(pyridin-3-yl)prop-2-en-1-imine (5d). A solution of 0.454 g (2 mmol) of oxime **7a** in 7 mL of anhydrous DMF was cooled with an ice–salt bath, and 0.120 g (3 mmol) of 60% sodium hydride was added under argon. The mixture was stirred for 30 min, 0.34 mL (2.5 mmol) of hexyl bromide was added, and the mixture was stirred for 18 h at room temperature and for 2.5 h at 60°C. The mixture was cooled, poured into ice water (50 mL), and extracted with diethyl ether. The extract was washed with 3 portions of water and 4 portions of 15% aqueous KOH, dried over Na_2SO_4, and evaporated. The residue was purified by flash chromatography on silica gel (5–40 μm) using first methylene chloride (to remove DMF and hexene) and then methylene chloride–ethyl acetate (gradient elution). Yield 0.427 g (65.5%), *E*/*Z* = 80:20, yellow oil. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.86 t (3H, CH_3, *J* = 6.9), 1.15–1.34 m [6H, $CH_3(CH_2)_3$], 1.62 m (2H, CH_2CH_2O), 4.04–4.30 m (2H, CH_2O), 6.36 d (0.20H, 3′-H, *Z*, *J* = ^{3}16.5), 6.67 d (0.80H, 3′-H, *E*, ^3J = 16.1), 6.93–7.51 m (6H, FC_6H_4, 5-H, 2′-H), 7.64 d (0.20H, 4-H, *Z*, *J* = 8.1) 7.73 d (0.80H, 4-H, *E*, *J* = 8.1), 8.51 s (1H, 2-H), 8.66 d (1H, 6-H, *J* = 4.8). Found, %: C 73.55; H 7.14; N 8.54. $C_{20}H_{23}FN_2O$. Calculated, %: C 73.42; H 7.19; N 8.48.

Compounds **5e**–**5i** were synthesized in a similar way.

(1*EZ*,2*E*)-3-(4-Chlorophenyl)-*N*-hexyloxy-1-(pyridin-3-yl)prop-2-en-1-imine (5e). Yield 0.489 g (71.4%), *E*/*Z* = 64:36, yellow oil. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0,81–0.95 m (3H, CH_3), 1.19–1.39 m [6H, $CH_3(CH_2)_3$], 1.55–1.84 m (2H, CH_2CH_2O), 4.06–4.29 m (2H, CH_2O), 6.35 d and 7.05 d (0.72H, CH=CH, *Z*, ^3J = 16.5), 6.72 d and 7.54 d (1.28H, CH=CH, *E*, ^3J = 16.9), 7.27–7.44 m (5H, 5-H, H_{arom}), 7.82 d (0.36H, 4-H, *Z*, *J* = 8.1), 8.35 d (0.64H, 4-H, *E*, *J* = 8.1), 8.55 s (1H, 2-H), 8.65 d (1H, 6-H, *J* = 5.0). Found, %: C 70.02; H 6.80; N 8.15. $C_{20}H_{23}ClN_2O$. Calculated, %: C 69.68; H 6.88; N 8.03.

(1*EZ*,2*E*)-3-(4-Bromophenyl)-*N*-hexyloxy-1-(pyridin-3-yl)prop-2-en-1-imine (5f). Yield 0.579 g (74.9%), *E*/*Z* = 60:40, yellow oil. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.72–0.96 m (3H, CH_3), 1.00–1.48 m [6H, $CH_3(CH_2)_3$], 1.50–1.83 m (2H, CH_2CH_2O), 4.05–4.30 m (2H, CH_2O), 6.44 d and 7.15 d (0.8H, CH=CH, *Z*, ^3J = 16.5), 6.76 d (0.60H, 2′-H, *E*, ^3J = 16.9), 7.15–7.66 m [5.60H, 2′-H (*E*), 5-H, H_{arom}], 7.85 d (0.40H, 4-H, *Z*, *J* = 8.1), 8.20 d (0.60H, 4-H, *E*, *J* = 7.8), 8.55 s (1H, 2-H), 8.66 d (1H, 6-H, *J* = 5.7). Found, %: C 61.98; H 6.03; N 7.21. $C_{20}H_{23}BrN_2O$. Calculated, %: C 62.15; H 6.13; N 7.00.

(1*EZ*,2*E*)-*N*-Cyclohexyloxy-3-(4-fluorophenyl)-1-(pyridin-3-yl)prop-2-en-1-imine (5g). Yield 0.088 g (13.6%), *E*/*Z* = 45:55 (yellow oil). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.79–0.94 m and 1.13–1.68 m [10H, $(CH_2)_5$], 4.05–4.29 m (1H, CHO), 6.36 d and 7.06 d (1.10H, CH=CH, *Z*, ^3J = 16.5), 6.68 d (0.45H, 2′-H, *E*, *J* = 17.3), 7.14–7.74 m [6.00H, 2′-H (*E*), 4-H (*Z*), 5-H, H_{arom}), 7.80 d (0.45H, 4-H, *E*, *J* = 8.0), 8.57 s (1H, 2-H), 8.66 d (1H, 6-H, *J* = 4.0). Found, %: C 74.01; H 6.56; N 8.62. $C_{20}H_{21}FN_2O$. Calculated, %: C 73.98; H 6.70; N 8.48.

(1*EZ*,2*E*)-3-(4-Chlorophenyl)-*N*-cyclohexyloxy-1-(pyridin-3-yl)prop-2-en-1-imine (5h). Yield 0.215 g (31.5%), *E*/*Z* = 55:45, yellow oil. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.15–1.67 m and 1.71–1.92 m [10H, $(CH_2)_5$], 4.10–4.30 m (1H, CHO), 6.36 d and 7.05 d (0.90H, CH=CH, *Z*, ^3J = 16.5), 6.67 d and 7.56 d (1.10H, CH=CH, *E*, ^3J = 16.9), 7.27–7.42 m (5H, 5-H, H_{arom}), 7.83 d (0.45H, 4-H, *Z*, *J* = 7.7), 8.35 d (0.55H, 4-H, *E*, *J* = 8.0), 8.55 s (1H, 2-H), 8.58–

8.67 m (1H, 6-H). Found, %: C 70.45; H 6.25; N 8.20. $C_{20}H_{21}ClN_2O$. Calculated, %: C 70.12; H 6.44; N 8.01.

(1*EZ*,2*E*)-3-(4-Bromophenyl)-*N*-cyclohexyloxy-1-(pyridin-3-yl)prop-2-en-1-imine (5i). Yield 0.149 g (19.3%), *E*/*Z* = 15:85, yellow oil. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.21–1.72 m and 1.74–1.87 m [10H, $(CH_2)_5$], 4.07–4.30 m (1H, CHO), 6.43 d and 7.15 d (1.90H, CH=CH, *Z*, *J* = 16.5), 6.67 d (0.15H, 2′-H, *E*, *J* = 16.9), 7.32–7.71 m [5.15H, 2′-H (*E*), 5-H, H_{arom}], 7.77 d (0.85H, 4-H, *Z*, *J* = 7.7), 7.85 d (0.15H, 4-H, *E*, *J* = 8.0), 8.52–8.71 m (1H, 6-H), 8.75 s (1H, 2-H). Found, %: C 62.31; H 5.53; N 7.25. $C_{20}H_{21}BrN_2O$. Calculated, %: C 61.97; H 5.71; N 7.00.

CONFLICT OF INTERESTS

The authors indicate the absence of conflict of interests.

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