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Synthesis of Alkyl Nitrones by Reaction of Aldehyde and Ketone Oximes with α,β-Unstaturated Esters in the Presence of Lewis Acid*

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Abstract—Reactions of pyridinecarbaldehyde oximes, methyl pyridyl ketone oximes, furfural oxime, cinnamaldehyde oxime, and crotonaldehyde oxime with acrylic and methacrylic acid esters in the presence of a Lewis acid catalyst at room temperature followed the conjugate addition pattern to give the corresponding alkyl nitrones in good yield. The best yields were obtained using a 1 : 1 mixture of CdI_2 and $BF_3 \cdot Bu_2O$ as catalyst.

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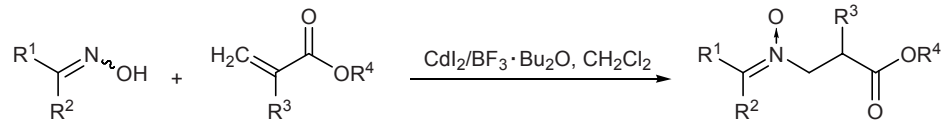
Alkylation of aldehyde and ketone oximes was studied in sufficient detail, and a number of physiologically and biologically active compounds were thus synthesized [1–6]. It is well known that compounds containing a pyridine ring are used as starting materials in the preparation of many medicines [7–13]. We previously synthesized in good yields some glycol ethers and halohydrins by alkylation of oximes containing hydroxy groups and unsaturated bonds with mono- and dihalohydrins in basic medium in the presence of phase-transfer catalyst [14–16]. Reactions of oximes with various alkylating agents provide one of the most convenient methods for generation of nitrones [17, 18].

In the present paper we report on the alkylation of some pyridinecarbaldehyde and methyl pyridyl ketone oximes with α,β-unsaturated carbonyl compounds (acrylic and methacrylic acid esters) in the presence of Lewis acid catalyst. Pyridine-2-carbaldehyde oxime (**Ia**) was allowed to react with methyl acrylate (**IIa**) in methylene chloride for 7 h at room temperature in the presence of a mixed Lewis acids catalyst, $CdI_2/BF_3 \cdot Bu_2O$ (1 : 1). The corresponding 1 : 1 adduct **III** was isolated in a quantitative yield and was characterized by analytical and spectral data (Scheme 1, Table 1). As unsaturated carbonyl component we also used methyl methacrylate (**IIb**), ethyl acrylate (**IIc**), ethyl methac-

Scheme 1.



Ia–Ih **IIa–IIe** **III–XVII**

I, R^1 = H, R^2 = (*Z*)-pyridin-2-yl (**a**), (*E*)-pyridin-3-yl (**b**), (*E*)-pyridin-4-yl (**c**); R^1 = Me, R^2 = (*E*,*Z*)-pyridin-2-yl (**d**), (*E*,*Z*)-pyridin-4-yl (**e**); R^1 = H, R^2 = 2-furyl (**f**), (*E*)-PhCH=CH (**g**), (*E*)-MeCH=CH (**h**); **II**, R^3 = H, R^4 = Me (**a**); R^3 = R^4 = Me (**b**); R^3 = H, R^4 = Et (**c**); R^3 = Me, R^4 = Et (**d**); R^3 = H, R^4 = *i*-Pr (**e**); **III**, R^1 = R^3 = H, R^2 = (*Z*)-pyridin-2-yl, R^4 = Me; **IV**, R^1 = H, R^2 = (*Z*)-pyridin-2-yl, R^3 = R^4 = Me; **V**, R^1 = R^3 = H, R^2 = (*Z*)-pyridin-2-yl, R^4 = Et; **VI**, R^1 = R^3 = H, R^2 = (*E*)-pyridin-3-yl, R^4 = Me; **VII**, R^1 = H, R^2 = (*E*)-pyridin-3-yl, R^3 = Me, R^4 = Et; **VIII**, R^1 = R^3 = H, R^2 = (*E*)-pyridin-4-yl, R^4 = Me; **IX**, R^1 = H, R^2 = (*E*)-pyridin-4-yl, R^3 = R^4 = Me; **X**, R^1 = R^3 = R^4 = Me, R^2 = (*E*,*Z*)-pyridin-2-yl; **XI**, R^1 = Me, R^2 = (*E*,*Z*)-pyridin-4-yl, R^3 = H, R^4 = *i*-Pr; **XII**, R^1 = Me, R^2 = (*E*,*Z*)-pyridin-4-yl, R^3 = H, R^4 = Et; **XIII**, R^1 = R^3 = H, R^2 = 2-furyl, R^4 = Me; **XIV**, R^1 = H, R^2 = 2-furyl, R^3 = Me, R^4 = Et; **XV**, R^1 = R^3 = H, R^2 = (*E*)-PhCH=CH, R^4 = Me; **XVI**, R^1 = H, R^2 = (*E*)-PhCH=CH, R^3 = R^4 = Me; **XVII**, R^1 = R^3 = H, R^2 = (*E*)-MeCH=CH, R^4 = Et.

* The text was submitted by the authors in English.

Table 1. Conjugate addition of oximes **Ia**–**Ih** to α,β-unsaturated esters **IIa**–**IIe** in the presence of $CdI_2/BF_3 \cdot Bu_2O$ as catalyst (50 mol %/50 mol %)

Oxime	Ester (equiv)	Solvent	Temperature	Time, h	Product (yield, %)
Ia	**IIa** (2.0)	CH_2Cl_2	Room temperature	7	**III** (88)
Ia	**IIb** (1.5)	Benzene	Reflux	24	**IV** (100)
Ia	**IIc** (1.5)	CH_2Cl_2	Room temperature	8	**V** (65)
Ib	**IIa** (1.5)	Benzene	Room temperature	7	**VI** (78)
Ib	**IId** (1.5)	CH_2Cl_2	Reflux	24	**VII** (84)
Ic	**IIa** (2.0)	Benzene	Room temperature	6	**VIII** (100)
Ic	**IIb** (1.5)	CH_2Cl_2	Reflux	24	**IX** (100)
Id	**IIb** (2.2)	Benzene	Reflux	24	**X** (100)
Ie	**IIe** (2.0)	CH_2Cl_2	Room temperature	24	**XI** (62)
Ie	**IIc** (1.8)	Benzene	Room temperature	24	**XII** (48)
If	**IIa** (1.3)	DMF	Room temperature	6	**XIII** (88)
If	**IId** (1.2)	DMF	Reflux	24	**XIV** (74)
Ig	**IIa** (1.0)	CH_2Cl_2	Room temperature	7	**XV** (100)
Ig	**IIb** (1.3)	CH_2Cl_2	Reflux	24	**XVI** (100)
Ih	**IIc** (1.0)	CH_2Cl_2	Room temperature	8	**XVII** (100)

rylate (**IId**), and isopropyl acrylate (**IIe**), which showed quite different reactivities (Table 1). Methyl acrylate (**IIa**) turned out to be the most reactive, whereas the reactions with methacrylic acid esters **IIb** and **IId** required heating in boiling benzene to complete the process. Methyl pyridyl ketone oximes, namely (*E*,*Z*)-1-(pyridin-2-yl)ethanone oxime (**Id**) and (*E*,*Z*)-1-(pyridin-4-yl)ethanone oxime (**Ie**), reacted with esters **IIb**, **IIc**, and **IIe** at an appreciably lower rate.

Unlike the data of [19], ketone oximes showed lower reactivity toward α,β-unsaturated esters, though the reactions described in [19] were carried out at elevated temperature. The formation of nitrones was usually fairly slow at room temperature without Lewis acid catalyst. On the other hand, (*E*)-cinnamaldehyde oxime (**Ig**) as one of the most reactive aldehyde oximes reacted with ester **IIb** in the absence of Lewis acid. The reactions of **Ig** and **Ih** with acrylates **IIa** and **IIc**, respectively, smoothly occurred at room temperature without a catalyst to give the corresponding nitrones **XV** and **XVII**.

Table 2. Lewis acid catalysis of the reaction of (*Z*)-pyridine-2-carbaldehyde oxime (**Ia**) with methyl acrylate (**IIa**) in methylene chloride at room temperature

Lewis acid (amount, mol %)	Time, h	Yield of **III**, %
CdI_2 (50)	42	18
CdI_2 (100)	8	38[a]
$BF_3 \cdot Bu_2O$ (50)	42	19
$BF_3 \cdot Bu_2O$ (100)	7	42[b]
$CdI_2/BF_3 \cdot Bu_2O$ (50/50)	7	68

[a] The reaction was accompanied by formation of the corresponding cycloadduct as by-product.

[b] Incomplete conversion.

The presence of mixed Lewis acid catalyst ($CdI_2/BF_3 \cdot Bu_2O$) is essential to ensure high yields of nitrones. In the reaction of **Ia** with **IIa** catalyzed by an equimolar amount of CdI_2, acceleration of not only the nitrone formation step but also of undesired dipolar cycloaddition step was observed, and the product was a mixture of nitrone **III** and the corresponding cycloadduct (Table 2). The reaction in the presence of an equimolar amount of $BF_3 \cdot Bu_2O$ was characterized by low conversion, and both starting compounds were partially recovered from the reaction mixture. Mixed Lewis acid catalyst consisting of equimolar amounts of CdI_2 and $BF_3 \cdot Bu_2O$ effectively mediated the nitrone formation step with high to quantitative yield (Table 1). The reason why mixed Lewis acid is particularly effective remains so far unclear. Heating should be avoided in the reactions with acceptors other than methyl methacrylate (**IIb**) and ethyl methacrylate (**IId**), otherwise the nitrones formed undergo serious decomposition to give complex mixtures of products.

The procedure for the isolation of nitrones is also critical: usual treatment of the reaction mixture with water is inappropriate. Alkyl nitrones are polar sub-

stances that are readily soluble in water; therefore, aqueous treatment may lead to considerable loss in the product yield. The following procedure is recommended: after the reaction is complete (according to the TLC data), the reaction mixture is filtered through a short silica gel column. If additional purification is necessary, repeated chromatography is performed.

Thus Lewis acid-catalyzed conjugate addition of aldehyde and ketone oximes to α,β-unsaturated esters at room temperature provides a convenient synthetic route to alkyl nitrones.

EXPERIMENTAL

All chemicals were purchased from Merck and Fluka and were used without additional purification. The melting points were determined on a Buchi capillary melting point apparatus and are uncorrected. The IR spectra were recorded on a Pye Unicam SP 1025 spectrometer. The ^{1}H NMR spectra were recorded from solutions in $CDCl_3$ on a Bruker Avance DPX instrument using tetramethylsilane as reference. The elemental compositions were determined on a Carlo Erba 1106 automatic CHN analyzer. Analytical TLC was performed on Merck 60 Kieselgel F 254 silica gel plates; spots were visualized under UV light. Preparative chromatography was performed using Merck 60 Kieselgel (0.040–0.063 mm).

General procedure for the alkylation of oximes. Acrylate **IIa**–**IId**, 1.5 or 2 equiv, was added to a solution of pyridinecarbaldehyde oxime **Ia**–**Ic**, 25 mmol, and Lewis acid catalyst (CdI_2/$BF_3 \cdot OBu_2$), 25 mmol, in 25 ml of methylene chloride unless otherwise stated (see Table 1). The mixture was stirred under the conditions indicated in Table 1, and the progress of the reaction was monitored by TLC. When the reaction was complete, the mixture was treated with 10 ml of methanol and evaporated, the residue was dissolved in methylene chloride, and the solution was subjected to column chromatography on silica gel or aluminum oxide using ethyl acetate–acetone (3 : 1) as eluent. Compounds **III**–**IX** were additionally recrystallized from petroleum ether or chloroform.

Methyl 3-[(*Z*)-oxido(pyridin-2-ylmethylidene)amino]propanoate (III). mp 130–131°C. IR spectrum, ν, cm^{-1}: 2355, 1780, 1762, 1694, 1422, 1400, 1380, 1362, 1344, 1275, 1191, 1120, 1043, 962, 940, 825, 758, 692. ^{1}H NMR spectrum, δ, ppm: 2.44 t (2H, CH_2CO), 3.58 s (3H, OCH_3), 3.94 t (2H, NCH_2), 7.84 d (1H, CH=N), 7.02–8.63 m (4H, pyridine). Found, %: C 57.54; H 5.76; N 13.36. $C_{10}H_{12}N_2O_3$. Calculated, %: C 57.68; H 5.81; N 13.45.

Methyl 2-methyl-3-{(*Z*)-oxido[1-(pyridin-2-yl)methylidene]amino}propanoate (IV). mp 118–119°C. IR spectrum, ν, cm^{-1}: 2345, 1768, 1756, 1694, 1422, 1410, 1380, 1340, 1270, 1120, 962, 820, 752, 684. ^{1}H NMR spectrum, δ, ppm: 1.15 d (3H, C**H**$_3$CH), 2.49 m (1H, C**H**CH$_3$), 3.58 s (3H, OCH_3), 3.93 t (2H, NCH_2), 7.91 s (1H, CH=N), 7.42–8.62 m (4H, pyridine). Found, %: C 59.33; H 6.49; N 12.48. $C_{11}H_{14}N_2O_3$. Calculated, %: C 59.45; H 6.35; N 12.60.

Ethyl 3-[(*Z*)-oxido(pyridin-2-ylmethylidene)amino]propanoate (V). mp 134–135°C. IR spectrum, ν, cm^{-1}: 2338, 1762, 1750, 1688, 1418, 1380, 1272, 1120, 962, 820, 752, 680. ^{1}H NMR spectrum, δ, ppm: 1.22 t (3H, CH_3), 2.42 t (2H, CH_2CO), 3.94 d (2H, NCH_2), 4.09 q (2H, OCH_2), 7.87 s (1H, CH=N), 7.39–8.64 m (4H, pyridine). Found, %: C 59.27; H 6.46; N 12.43. $C_{11}H_{14}N_2O_3$. Calculated, %: C 59.45; H 6.35; N 12.60.

Methyl 3-[(*E*)-oxido(pyridin-3-ylmethylidene)amino]propanoate (VI). mp 128–129°C. IR spectrum, ν, cm^{-1}: 2325, 1760, 1745, 1680, 1410, 1384, 1270, 1120, 964, 820, 750, 680. ^{1}H NMR spectrum, δ, ppm: 2.43 t (2H, CH_2CO), 3.58 s (3H, OCH_3), 3.94 t (2H, NCH_2), 7.62 s (1H, CH=N), 7.22–8.74 m (4H, pyridine). Found, %: C 57.52; H 5.66; N 13.31. $C_{10}H_{12}N_2O_3$. Calculated, %: C 57.68; H 5.81; N 13.45.

Ethyl 2-methyl-3-[(*E*)-oxido(pyridin-3-ylmethylidene)amino]propanoate (VII). mp 126–127°C. IR spectrum, ν, cm^{-1}: 2316, 1758, 1740, 1665, 1402, 1380, 1276, 1120, 960, 815, 750, 675. ^{1}H NMR spectrum, δ, ppm: 1.12 d (3H, CHC**H**$_3$), 1.24 t (3H, C**H**$_3$CH$_2$), 2.42 m (1H, C**H**CH$_3$), 3.92 d (2H, NCH_2), 4.08 q (2H, OCH_2), 7.87 s (1H, CH=N), 7.76–8.82 m (4H, pyridine). Found, %: C 61.14; H 6.98; N 11.72. $C_{12}H_{16}N_2O_3$. Calculated, %: C 61.00; H 6.84; N 11.85.

Methyl 3-[(*E*)-oxido(pyridin-4-ylmethylidene)amino]propanoate (VIII). mp 151–152°C. IR spectrum, ν, cm^{-1}: 2312, 1780, 1742, 1660, 1400, 1384, 1270, 1120, 965, 812, 755, 670. ^{1}H NMR spectrum, δ, ppm: 2.43 t (2H, CH_2CO), 3.58 s (3H, OCH_3), 3.94 t (2H, NCH_2), 8.02 d (1H, CH=N), 7.52–8.76 m (4H, pyridine). Found, %: C 57.48; H 5.96; N 13.55. $C_{10}H_{12}N_2O_3$. Calculated, %: C 57.67; H 5.81; N 13.44.

Methyl 2-methyl-3-[(*E*)-oxido(pyridin-4-ylmethylidene)amino]propanoate (IX). mp 146–147°C. IR spectrum, ν, cm^{-1}: 2325, 1745, 1720, 1665, 1400, 1375, 1275, 1120, 962, 810, 750, 665. ^{1}H NMR spectrum, δ, ppm: 1.18 d (3H, CHC**H**$_3$), 2.48 m (1H,

C**H**CH_3), 3.58 s (3H, OCH_3), 3.93 t (2H, NCH_2), 7.81 s (1H, CH=N), 7.48–8.76 m (pyridine). Found, %: C 59.58; H 6.54; N 12.47. $C_{12}H_{14}N_2O_3$. Calculated, %: C 59.45; H 6.36; N 12.61.

Nitrones X–XII (*general procedure***).** Ester **IIb**, **IIc**, or **IIe**, 25 mmol, was added to a solution of methyl pyridyl ketone oxime **Id** or **Ie**, 10 mmol, and an equivalent amount of Lewis acid catalyst (CdI_2/$BF_3 \cdot OBu_2$) in 20 ml of benzene or CH_2Cl_2. The mixture was stirred under the conditions indicated in Table 1 and treated with 15 ml of methanol, the solvent was removed on a rotary evaporator, the residue was dissolved in acetone or ethyl acetate, and the product was isolated by column chromatography and additionally recrystallized from petroleum ether or chloroform.

Methyl 2-methyl-3-{(*E*,*Z*)-oxido[1-(pyridin-2-yl)ethylidene]amino}propanoate (X). mp 138°C. IR spectrum, ν, cm^{-1}: 2318, 1748, 1716, 1660, 1415, 1372, 1270, 1122, 960, 812, 755, 662. ^{1}H NMR spectrum, δ, ppm: 1.14 d (3H, C**H**$_3$CH), 2.10 s (3H, CH_3C=N), 2.52 m (1H, CH_3C**H**), 3.88 m (2H, NCH_2), 4.05 s (3H, OCH_3), 6.86–8.54 m (4H, pyridine). Found, %: C 62.18; H 7.42; N 11.04. $C_{13}H_{18}N_2O_3$. Calculated, %: C 62.37; H 7.23; N 11.19.

Isopropyl 3-{(*E*,*Z*)-oxido[1-(pyridin-4-yl)ethylidene]amino}propanoate (XI). mp 158–159°C. IR spectrum, ν, cm^{-1}: 2314, 1745, 1710, 1662, 1410, 1370, 1280, 1120, 966, 814, 750, 660. ^{1}H NMR spectrum, δ, ppm: 1.14 d [6H, CH(C**H**$_3$)$_2$], 2.42 t (2H, CH_2CO), 3.43 s (3H, CH_3C=N), 3.86 t (2H, NCH_2), 5.02 m [1H, C**H**$(CH_3)_2$], 6.70–8.42 m (4H, pyridine). Found, %: C 62.21; H 7.34; N 11.32. $C_{13}H_{18}N_2O_3$. Calculated, %: C 62.37; H 7.23; N 11.19.

Ethyl 3-{(*E*,*Z*)-oxido[1-(pyridin-4-yl)ethylidene]amino}propanoate (XII). mp 162°C. IR spectrum, ν, cm^{-1}: 2312, 1746, 1712, 1660, 1414, 1368, 1282, 1120, 965, 812, 755, 665. ^{1}H NMR spectrum, δ, ppm: 1.28 t (3H, CH_3), 2.42 t (2H, CH_2CO), 3.36 s (2H, CH_2C=N), 3.86 t (2H, NCH_2), 4.08 q (2H, OCH_2), 6.70–8.42 m (4H, pyridine). Found, %: C 60.86; H 6.74; N 11.97. $C_{12}H_{16}N_2O_3$. Calculated, %: C 61.01; H 6.83; N 11.86.

Nitrones XIII and XIV (*general procedure***).** Ester **IIa** or **IId**, 45 mmol, was added to a solution of 20 mmol of furfural oxime (**If**) and an equivalent amount of Lewis acid catalyst (CdI_2/$BF_3 \cdot Bu_2O$) in 20 ml of anhydrous dimethylformamide. The mixture was stirred under the conditions indicated in Table 1 and treated with 15 ml of methanol. The solvent was evaporated, the residue was dissolved in methylene chloride, and the solution was subjected to column chromatography. The product was additionally purified by recrystallization from petroleum ether or DMSO.

Methyl 3-{[(furan-2-yl)methylidene](oxido)amino}propanoate (XIII). mp 112–113°C. IR spectrum, ν, cm^{-1}: 2310, 1755, 1710, 1665, 1410, 1362, 1280, 1120, 962, 812, 750, 660. ^{1}H NMR spectrum, δ, ppm: 2.42 m (2H, CH_2CO), 3.58 s (3H, OCH_3), 3.92 t (2H, NCH_2), 6.63–6.66 m (2H, furan), 7.42 d (1H, CH=N), 7.62 d (1H, furan). Found, %: C 54.65; H 5.88; N 7.23. $C_9H_{11}NO_4$. Calculated, %: C 54.83; H 5.60; N 7.11.

Ethyl 2-methyl-3-{[(furan-2-yl)methylidene](oxido)amino}propanoate (XIV). mp 119–120°C. IR spectrum, ν, cm^{-1}: 2314, 1750, 1714, 1662, 1408, 1350, 1285, 1125, 965, 814, 755, 665. ^{1}H NMR spectrum, δ, ppm: 1.13 d (3H, CHC**H**$_3$), 1.32 t (3H, CH_2C**H**$_3$), 2.45 m (1H, C**H**CH_3), 3.92 m (2H, NCH_2), 4.12 q (2H, OCH_2), 6.56–6.59 m (2H, furan), 7.54 d (1H, furan). Found, %: C 54.65; H 5.88; N 7.23. $C_{11}H_{15}NO_4$. Calculated, %: C 58.65; H 6.75; N 6.21.

Nitrones XV–XVII (*general procedure***).** Ester **IIa–IIc**, 20 mmol, was added to a solution of 20 mmol of (*E*)-cinnamaldehyde oxime (**Ig**) or (*E*)-crotonaldehyde oxime (**Ih**) and an equivalent amount of CdI_2/$BF_3 \cdot Bu_2O$ in 25 ml of CH_2Cl_2. The mixture was stirred under the conditions indicated in Table 1 and was then treated as described above for nitrones **III–IX**.

Methyl 3-{oxido[(2*E*)-3-phenylprop-2-en-1-ylidene]amino}propanoate (XV). mp 150–151°C. IR spectrum, ν, cm^{-1}: 2295, 1755, 1710, 1660, 1410, 1382, 1260, 1120, 960, 812, 752, 665. ^{1}H NMR spectrum, δ, ppm: 2.44 t (2H, CH_2CO), 3.58 s (3H, OCH_3), 3.90 d (2H, NCH_2), 7.10–7.12 m (2H, CH=CH), 7.50 m (1H, CH=N), 7.24–7.53 m (5H, H_{arom}). Found, %: C 66.76; H 6.63; N 6.13. $C_{13}H_{15}NO_3$. Calculated, %: C 66.93; H 6.48; N 6.02.

Methyl 2-methyl-3-{oxido[(2*E*)-3-phenylprop-2-en-1-ylidene]amino}propanoate (XVI). mp 142–143°C. IR spectrum, ν, cm^{-1}: 2285, 1750, 1720, 1665, 1492, 1380, 1265, 1125, 965, 812, 750, 660. ^{1}H NMR spectrum, δ, ppm: 1.19 d (3H, C**H**$_3$CH), 2.46 m (1H, CH_3C**H**), 3.56 s (3H, OCH_3), 3.90 d (2H, NCH_2), 6.92–7.20 d (2H, CH=CH), 7.30–7.50 m (5H, H_{arom}), 7.62 m (1H, CH=N). Found, %: C 68.11; H 6.85; N 5.56. $C_{14}H_{17}NO_3$. Calculated, %: C 68.01; H 6.94; N 5.66.

Ethyl 3-[(2*E*)-but-2-en-1-ylidene(oxido)amino]-propanoate (XVII). mp 165–167°C. IR spectrum, ν, cm^{-1}: 2270, 1760, 1715, 1662, 1495, 1384, 1260, 1120, 960, 815, 755, 668. ^{1}H NMR spectrum, δ, ppm: 1.22 t

(3H, CH_3), 1.81 d (3H, CH_3C=), 2.36 t (2H, CH_2CO), 3.77 t (2H, NCH_2), 4.07 m (2H, OCH_2), 6.17–6.36 m (2H, CH=CH), 7.52 d (1H, CH=N). Found, %: C 58.12; H 8.31; N 7.40. $C_9H_{15}NO_3$. Calculated, %: C 58.37; H 8.15; N 7.55.

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