DOI 10.1007/s10593-015-1744-z

Chemistry of Heterocyclic Compounds **2015**, *51*(7), 621–629

Dedicated to the memory of Professor A. V. Butin, friend, colleague, teacher

A simple method for the synthesis of furfuryl ketones and furylacetic acid derivatives

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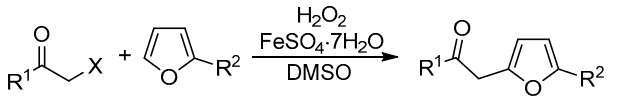
Translated from Khimiya Geterotsiklicheskikh Soedinenii, 2015, *51*(7), 621–629

Submitted June 5, 2015
Accepted June 15, 2015



X = I, SC(S)OEt;
R^1 = Ar, NH_2, NHAr, OAr; R^2 = H, Alk, Ar

A simple preparative method has been developed for the synthesis of aryl(furfuryl) ketones, amides, and furylacetic acid esters, based on radical alkylation of furan derivatives at the α-position with *O*-ethyl(phenacyl)xanthogenates and phenacyl iodides in the presence of Fenton's reagent (H_2O_2/$FeSO_4$·$7H_2O$) in DMSO. The range of applicability and mechanisms for the formation of major and side products have been considered.

Keywords: furans, furylacetamides, furfuryl ketones, esters of furylacetic acids, radical alkylation.

One of the key directions for the development of organic synthesis in recent years is the emphasis on renewable resources, especially biomass. The combination of purely chemical and also economic aspects has allowed to identify the most promising raw materials of biological origin that can be used for fine organic synthesis, denoted as molecular platforms.[1a] Important examples of such compounds are furan derivatives, which, on one hand, undergo reactions typical of aromatic compounds, while on the other hand can react as synthetic equivalents of alkenes, 1,3-dienes, enol ethers, and 1,4-diketones. Such a unique reactivity enables the application of furan molecular platforms for the preparation of practically useful products of furan series,[2] as well as other types of compounds.[3] In particular, furans containing an appropriate functional group may undergo intramolecular reactions with loss of aromaticity in the furan ring, producing other heterocycles.[4]

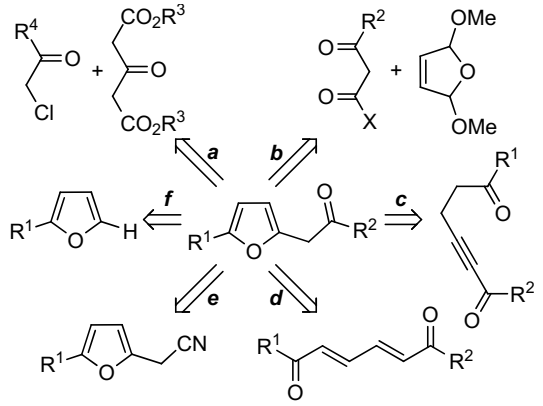
During our investigation of furan recyclization, we were interested in their β-carbonyl derivatives, in particular furfuryl ketones and derivatives of (2-furyl)acetic acids. The simultaneous presence of a furan ring, carbonyl group, and easily modified methylene linker in these molecules creates a significant potential for using such furans as synthetic building blocks.[5]

Two major types of methods for the synthesis of (2-furyl)-acetic acids and furfuryl ketones can be distinguished: a) reactions leading to the formation of furan ring, containing the required functional group, and b) functionalization of an existing furan derivative.[6] Thus, synthesis of furylacetic acids and their derivatives was accomplished by Feist–Benary condensation of acetonedicarboxylic acid ester with α-chlorocarbonyl compounds (Route ***a***, Scheme 1)[7] and related transformations.[8] Another method for the synthesis of these compounds is acid-catalyzed interaction of 1,3-dicarbonyl compounds with 2,5-dimethoxy-2,5-dihydrofurans[9] (Route ***b***, Scheme 1) or its equivalent, 1,2-diacylethylene.[10] Cyclizations of γ-(alkynyl) ketones[11] (Route ***c***, Scheme 1), 2-en-4-yn-1-ols,[12] 2,4-diene-1,6-diones[13] (Route ***d***, Scheme 1) and related processes are also important.[14] Among the methods for functionalization of substituted furans, we should mention the hydrolysis of (2-furyl)acetonitriles[15] (Route ***e***, Scheme 1), oxidation of the respective alcohols,[16] Suzuki reaction,[17] and other processes.[18]

The presence of furfuryl ketone fragment in a range of natural compounds that are of interest to medicinal chemistry motivates the development of new methods for the synthesis of furfuryl ketones and (2-furyl)acetic acids. The promise of direct functionalization of 2-substituted furans is based on their ready availability from furfural, a product of biomass processing (Route ***f***, Scheme 1).[1b]

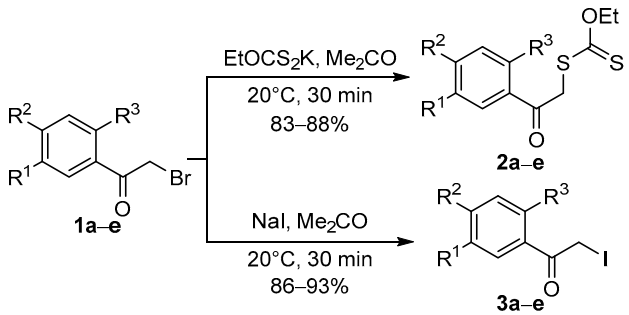
† Deceased

Scheme 1



Retrosynthetic analysis of
(2-furyl)acetic acid and furfuryl ketones

Scheme 2



1–**3** **a**–**c** $R^1 = R^3 = H$, **a** $R^2 = Br$, **b** $R^2 = NO_2$, **c** $R^2 = OMe$,
1–**3** **d**,**e** $R^1 = R^2 = OMe$, **d** $R^3 = H$, **e** $R^3 = NO_2$

However, the introduction of 2-oxoalkyl group in the furan ring *via* Friedel–Crafts reaction is limited to the use of glyoxylates,[19] α-acyl-α-chlorosulfides,[20] and related compounds, leading to α-substituted products. On the other hand, oxoalkylation of furan with radicals generated by oxidation of acetone or malonic ester with Mn(III) acetate was described in 1981 and gave moderate yields.[21] Esters of 2-(hetaryl)acetic acids, containing electron-withdrawing substituents at the C-5 atom, were obtained by using ethyl xanthogenacetate,[22] but this procedure is not generally applicable, for example, it is not suitable for the modification of 2-alkylfurans.[23] The Baciocchi method is more promising, where furan is alkylated with iodoacetic acid ester in DMSO at room temperature in the presence of Fenton's reagent ($H_2O_2/FeSO_4·7H_2O$), giving ethyl ester of (2-furyl)acetic acid in 65% yield.[23,24]

Our preliminary results show that using *O*-ethyl-(phenacyl)xanthogenates under these conditions for alkylation of 2-methylfuran allowed to obtain the corresponding 2-(phenacyl)furans in good yields.[25] In the current work, we present the complete results regarding radical alkylation of furan derivatives with phenacyl iodides and xanthogenates, as well as describe for the first time the preparation of phenyl esters and arylamides of furylacetic acid under the aforementioned conditions. The starting *O*-ethyl(phenacyl)xanthogenates **2** and phenacyl iodides **3** were obtained from readily available phenacyl bromides **1** (Scheme 2).

We started the study of furan alkylation by optimization of conditions for the model reaction between xanthogenate **2a** and 2-methylfuran (**4a**) (Scheme 3). We found that the maximum yield of the product was obtained by performing the reaction for 1 h with 1:8:4 molar ratio of **2a**:H_2O_2:**4a**. Using a smaller excess of furan **4a** relative to compound **2a** (1:2) resulted in a slightly lower yield of the final product.[25] On the other hand, increasing the excess of 2-methylfuran (**4a**) to 1:10 and above did not noticeably increase the yield of 2-(phenacyl)furan **5a**. A lower amount of peroxide was insufficient for complete conversion of xanthogenate **2a**, while increasing the amount of peroxide led to a decreased yield of product **5a**, apparently due to its oxidation with excess peroxide. Increased reaction time also resulted in accumulation of products from decomposition of 2-phenacylfuran **5a**. Besides that, we studied the possibility of alkylating furan **4a** with *O*-*tert*-butyl-, *O*-[2-(methylsulfanyl)ethyl]-, and *O*-[2-(dimethyl-amino)ethyl]xanthogenates, but found that the results of the reaction were not improved by using these relatively difficult to prepare reagents.

Under the optimized conditions we obtained a series of 2-(phenacyl)furans **5a**–**h** (Table 1). We should note that the formation of two by-products was always observed. These by-products were isolated in three cases, characterized by spectroscopy and elemental analysis, and assigned with the structure of sulfones **6a**,**e**,**g**, while 1,4-diaryl 1,4-diketones **7a**,**e**,**g** were obtained in smaller quantities (5–7%) (Scheme 3).

Nevertheless, good yields of products **5** were obtained both for 2-alkylfurans **4a**,**b** and furan itself (**4d**), as well as for 2-arylfuran **4c**. On the other hand, the introduction of electron-withdrawing substituent at the α-position of furan ring suppressed the alkylation, as in the case of

Scheme 3

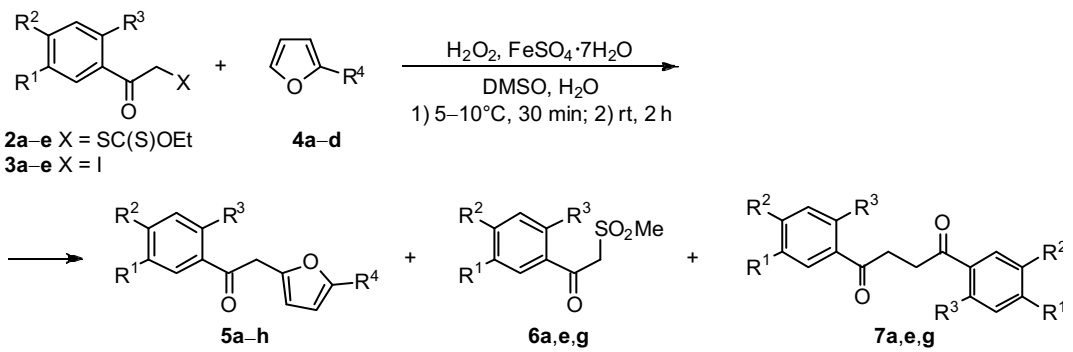


Table 1. Structures and yields of phenacylfurans **5a**–**h**, sulfones **6a**,**e**,**g**, and diketones **7a**,**e**,**g**

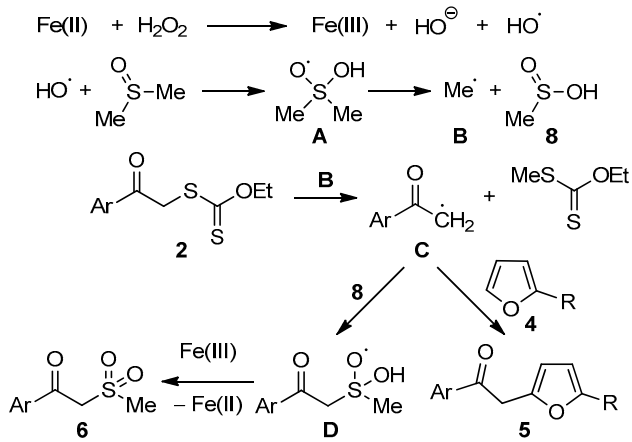
Xanthogenate or iodide	R^1	R^2	R^3	Furan	R^4	2-Phenacylfuran (Yield, %)	Sulfone (Yield, %)	Diketone (Yield, %)
2a	H	Br	H	**4a**	Me	**5a** (61)	**6a** (22)	**7a** (7)
3a	H	Br	H	**4a**	Me	**5a** (54)	–	–
2a	H	Br	H	**4b**	*t*-Bu	**5b** (58)	–	–
2a	H	Br	H	**4c**	4-ClC_6H_4	**5c** (55)	–	–
2a	H	Br	H	**4d**	H	**5d** (53)	–	–
2a	H	Br	H	**4e**	CO_2Et	–	**6a** (62)	–
2b	H	NO_2	H	**4a**	Me	**5e** (55)	**6e** (31)	**7e** (5)
3b	H	NO_2	H	**4a**	Me	**5e** (47)	–	–
2c	H	OMe	H	**4a**	Me	**5f** (58)	–	–
3c	H	OMe	H	**4a**	Me	**5f** (35)	–	–
2d	OMe	OMe	H	**4a**	Me	**5g** (64)	**6g** (25)	**7g** (5)
3d	OMe	OMe	H	**4a**	Me	**5g** (56)	–	–
2e	OMe	OMe	NO_2	**4a**	Me	**5h** (52)	–	–
3e	OMe	OMe	NO_2	**4a**	Me	**5h** (37)	–	–

xanthogenate **2a** reaction with ester **4e**, where the only product isolated in 62% yield was the sulfone **6a**. Analogous result was obtained for 2-acetylfuran. We should also note that iodides **3** gave somewhat lower yields of alkylation products, compared to the respective xanthogenates.

The proposed reaction mechanism is shown in Scheme 4. The first stage of this process is the well-known single-electron reduction of hydrogen peroxide in the presence of Fe(II) salts with the formation of a hydroxide ion and a hydroxyl radical. The high reactivity of hydroxyl radical may be moderated by using DMSO as solvent, increasing the reaction selectivity. Indeed, the use of other solvents (DMF, acetonitrile, 1,4-dioxane) gave significantly inferior results. Thus, the yield of phenacylfuran **5a** in DMF was merely 20%, while in acetonitrile and 1,4-dioxane this product was formed in trace amounts, detected only chromatographically. Hydroxyl radical is well known to react rapidly with DMSO, forming intermediate **A**, decomposition of which gives methyl radical **B** and methylsulfinic acid **8**.[26] The interaction of radical **B** with xanthogenate **2** is accompanied by the formation of phenacyl radical **C**, which reacts with furan **4**, giving compound **5**. The formation of sulfone **6** as by-product apparently can be explained by the interaction of radical **C** with methylsulfinic acid **8** that accumulates in the reaction mixture, leading to intermediate **D**. Oxidation of the latter with Fe(III) ions produces the sulfone **6**.

We also studied the possibility of using radical alkylation with xanthogenates in DMSO in the presence of Fenton's reagent for the synthesis of phenyl esters and amides of (5-methylfuran-2-yl)acetic acid. The starting xanthogenates **11a**–**i** were obtained in two stages – by acylation of amines **9a**–**g** and phenols **9h**,**i** with chloroacetyl chloride, followed by treatment of the obtained chlorides **10** with potassium xanthogenate in acetone. We found that, under analogous conditions to those used for preparation of phenacylfurans **5**, xanthogenates **11** alkylated 2-methylfuran (**4a**) in moderate yields, forming amides **12a**–**g** and phenyl esters **12h**,**i** (Scheme 5).

Scheme 4



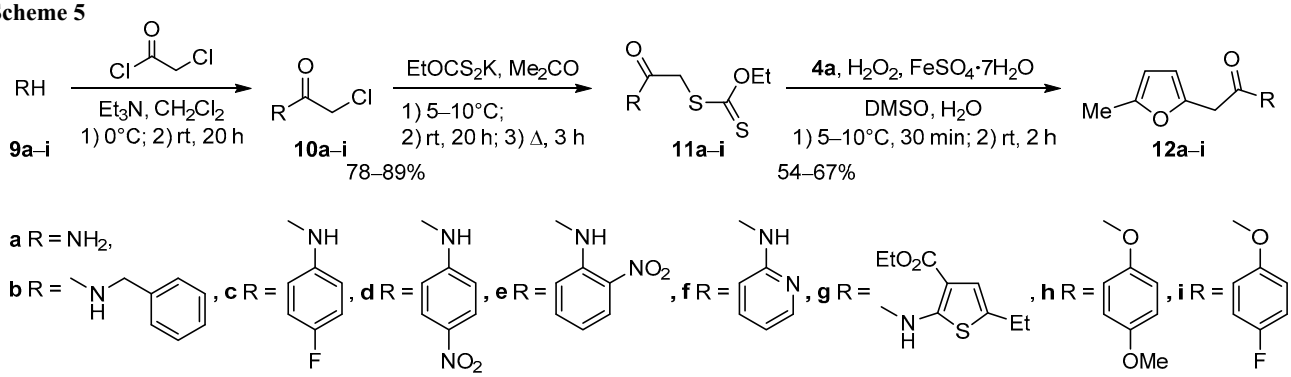
Despite moderate yields, the simplicity of this method for the synthesis of esters and amides of furylacetic acid and the availability of starting compounds are significant advantages, compared to the traditional acylation of less available furylacetic acids.

Thus, we have shown that alkylation of furans in the presence of Fenton's reagent (H_2O_2/$FeSO_4$·$7H_2O$) with the appropriate xanthogenates in DMSO at room temperature allowed to obtain various 2-oxoalkyl derivatives of furan in moderate yields. The reaction worked with xanthogenates of various structures and furans that contained alkyl or aromatic substituents, as well as unsubstituted furan. In contrast, furans with electron-withdrawing substituents did not participate in this reaction. It was shown that the use of phenacyl xanthogenates resulted in the formation of methyl-(phenacyl)sulfones and symmetrical 1,4-diaryl 1,4-diketones as by-products. A mechanism for the formation of both the major and minor reaction products has been postulated.

Experimental

IR spectra were recorded on FSM-1202 (compounds **2e**, **5b**–**d**,**g**,**h**, **11c**,**e**,**f**–**i**, **12b**,**c**,**e**–**i**) and Bruker Alpha FT-IR spectrometers (compounds **11d**, **2d**). ^1H and ^{13}C NMR

Scheme 5



spectra were acquired on Bruker DRX-300 (300 and 75 MHz, respectively), Bruker Avance 600 (600 and 150 MHz, respectively), and Bruker Avance III HD 400 (400 and 100 MHz respectively) instruments at room temperature in $CDCl_3$ or DMSO-d_6; the residual solvent protons were used as internal standard ($CDCl_3$: 7.26 ppm for ^1H nuclei, 77.16 ppm for ^{13}C nuclei; DMSO-d_6: 2.50 ppm for ^1H nuclei, 39.52 ppm for ^{13}C nuclei). Mass spectra were recorded on a Kratos MS-30 mass spectrometer, EI ionization (ionization voltage 70 eV, ionization chamber temperature 200°C) or a HEVO TQD ACQUITY UPLC-I chromato-mass spectrometer with electrospray ionization. Elemental analysis was performed on a vario MICRO cube CHNS analyzer. Melting points were determined on a Stuart SMP30 apparatus. TLC analysis was performed on Sorbfil plates. Reaction mixtures were purified by using KSK silica gel.

Phenacyl xanthogenates **2** were obtained according to a published procedure.[27]

***S*-[2-(4-Bromophenyl)-2-oxoethyl] *O*-ethyl dithiocarbonate (2a)**. Yield 88%, white powder, mp 84–86°C (3:1 petroleum ether–CH_2Cl_2) (mp 81–83°C[28]). ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.40 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 4.61 (2H, s, CH_2); 4.64 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 7.65 (2H, AA'BB' system, ^3J = 8.4, H Ar); 7.88 (2H, AA'BB' system, ^3J = 8.4, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.9; 43.5; 71.0; 129.2; 130.1 (2C); 132.3 (2C); 134.7; 191.6; 213.3.

***O*-Ethyl *S*-[2-(4-nitrophenyl)-2-oxoethyl] dithiocarbonate (2b)**. Yield 87%, white powder, mp 87–89°C (3:1 petroleum ether – CH_2Cl_2) (mp 88–89°C (petroleum ether–diethyl ether)[29]). ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.41 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 4.64 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 4.66 (2H, s, CH_2); 8.18 (2H, AA'BB' system, ^3J = 8.8, H Ar); 8.35 (2H, AA'BB' system, ^3J = 8.8, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.9; 43.6; 71.3; 124.1 (2C); 129.6 (2C); 140.6; 150.8; 191.5; 213.0.

***O*-Ethyl *S*-[2-(4-methoxyphenyl)-2-oxoethyl] dithiocarbonate (2c)**. Yield 83%, beige powder, mp 68–69°C (4:1 petroleum ether–EtOAc) (mp 67°C (hexane–EtOAc)[31]). ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.40 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 3.89 (3H, s, OCH_3); 4.62 (2H, s, CH_2); 4.65 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 6.97 (2H, AA'BB' system, ^3J = 8.8, H Ar); 8.00 (2H, AA'BB' system, ^3J = 8.8, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.9; 43.5; 55.7; 70.7; 114.2 (2C); 129.1; 131.0 (2C); 164.2; 191.0; 213.7.

***O*-Ethyl *S*-[2-(3,4-dimethoxyphenyl)-2-oxoethyl] dithiocarbonate (2d)**. Yield 84%, white powder, mp 75–77°C (3:1 petroleum ether–CH_2Cl_2) (mp 73–74°C (EtOH)[27]). ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.39 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 3.93 (3H, s, OCH_3); 3.95 (3H, s, OCH_3); 4.63 (2H, s, CH_2); 4.63 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 6.91 (1H, d, ^3J = 8.4, H Ar); 7.54 (1H, d, ^4J = 1.8, H Ar); 7.67 (1H, dd, ^3J = 8.4, ^4J = 1.8, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.9; 43.3; 56.2; 56.3; 70.8; 110.3; 110.7; 123.4; 129.1; 149.4; 154.0; 191.0; 213.7.

***O*-Ethyl *S*-[2-(4,5-dimethoxy-2-nitrophenyl)-2-oxoethyl] dithiocarbonate (2e)**. Yield 88%, light-yellow powder, mp 117–119°C (3:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 1721 (C=O), 1581, 1533, 1335, 1279, 1219, 1177, 1114, 1055, 1005. ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.38 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 3.97 (3H, s, OCH_3); 3.99 (3H, s, OCH_3); 4.43 (2H, s, CH_2); 4.59 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 6.89 (1H, s, H Ar); 7.64 (1H, s, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.8; 45.7; 56.7; 56.9; 71.2; 106.7; 110.0; 130.9; 138.5; 150.0; 154.2; 196.0; 213.7. Mass spectrum (EI), *m/z* (I_{rel}, %): 345 $[M]^+$ (29), 312 (39), 299 (32), 240 (23), 224 (35), 210 (100), 195 (52), 181 (51), 164 (38), 136 (71), 123 (40), 106 (43), 93 (62), 75 (55). Found, %: C 45.18; H 4.42; N 4.09. $C_{13}H_{15}NO_6S_2$. Calculated, %: C 45.21; H 4.38; N 4.06.

Phenacyl iodides 3 were obtained according to a published procedure.[30]

1-(4-Bromophenyl)-2-iodoethanone (3a). Yield 89%, colorless needles, mp 94–96°C (3:1 petroleum ether–EtOAc) (mp 95–96°C (6:1 hexane–EtOAc)[30]). ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 4.31 (2H, s, CH_2); 7.63 (2H, AA'BB' system, ^3J = 8.5, H Ar); 7.85 (2H, AA'BB' system, ^3J = 8.5, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 1.0; 129.2; 130.7 (2C); 132.4 (2C); 132.6; 191.9.

2-Iodo-1-(4-nitrophenyl)ethanone (3b). Yield 92%, yellow powder, mp 96–98°C (3:1 petroleum ether–EtOAc) (mp 93.5–94.5°C[32]). ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 4.39 (2H, s, CH_2); 8.14 (2H, AA'BB' system, ^3J = 8.7, H Ar); 8.33 (2H, AA'BB' system, ^3J = 8.7, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 0.8; 124.2 (2C); 130.2 (2C); 138.3; 150.9; 191.3.

2-Iodo-1-(4-methoxyphenyl)ethanone (3c). Yield 86%,

pale-yellow powder, mp 59–60°C (4:1 petroleum ether–EtOAc) (mp 58–59°C[32]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 3.88 (3H, s, OCH_3); 4.31 (2H, s, CH_2); 6.94 (2H, AA'BB' system, ^3J = 8.9, H Ar); 7.97 (2H, AA'BB' system, ^3J = 8.9, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 1.8; 55.7; 114.1 (2C); 126.5; 131.5 (2C); 164.1; 191.6.

2-Iodo-1-(3,4-dimethoxyphenyl)ethanone (3d). Yield 88%, pale-yellow powder, mp 66–67°C (3:1 petroleum ether–EtOAc) (mp 65.2–66.2°C (pentane–acetone)[33]). ^{1}H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 3.93 (3H, s, OCH_3); 3.95 (3H, s, OCH_3); 4.32 (2H, s, CH_2); 6.89 (1H, d, ^3J = 8.4, H Ar); 7.53 (1H, d, ^4J = 2.0, H Ar); 7.61 (1H, dd, ^3J = 8.4, ^4J = 2.0, H Ar). ^{13}C NMR spectrum (75 MHz, $CDCl_3$), δ, ppm: 1.4; 56.2; 56.3; 110.2; 111.1; 124.0; 126.7; 149.4; 154.0; 191.8.

2-Iodo-1-(4,5-dimethoxy-2-nitrophenyl)ethanone (3e). Yield 93%, yellow powder, mp 153–155°C (1:1 petroleum ether–CH_2Cl_2) (mp 138–141°C (EtOH) [34]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 4.00 (3H, s, OCH_3); 4.01 (3H, s, OCH_3); 4.20 (2H, s, CH_2); 6.92 (1H, s, H Ar); 7.66 (1H, s, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 6.9; 56.8; 57.0; 106.9; 112.1; 129.4; 138.3; 150.3; 154.4; 195.3.

Preparation of 2-(phenacyl)furans 5a–h, acetamides 12a–g, and carboxylates 12h,i (General method). A solution of xanthogenate **2a**–**e** or **11a**–**i** or iodide **3a**–**e** (10 mmol) and of furan **4a**–**d** (80 mmol of furans **4a**,**b**,12 mmol of furan **4c**, 200 mmol of furan **4d**) in DMSO (30 ml) was cooled (5–10°C) and treated with $FeSO_4 \cdot 7H_2O$ (1.52 g, 5 mmol). Then 34% aqueous H_2O_2 (2 ml, 20 mmol) was dropwise added. After adding H_2O_2, the reaction mixture was stirred for 30 min at 5–10°C, then for 2 h at room temperature (control by TLC). Once the reaction was complete, the mixture was poured into water (300 ml), extracted with ethyl acetate (4×50 ml), the combined extracts were washed with saturated aqueous NaCl solution, dried over anhydrous Na_2SO_4, and evaporated to dryness. The residue was separated by silica gel column chromatography with 4:1 petroleum ether–EtOAc as eluent (9:1 for compounds **5g**,**h**).

1-(4-Bromophenyl)-2-(5-methylfuran-2-yl)ethanone (5a).[25] Yield 1.70 g (61%), light-beige powder, mp 58–59°C (9:1 petroleum ether–EtOAc) (mp 58–59°C (petroleum ether–EtOAc)[25]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.25 (3H, s, CH_3); 4.20 (2H, s, CH_2); 5.90 (1H, d, ^3J = 3.0, H Fur); 6.08 (1H, d, ^3J = 3.0, H Fur); 7.60 (2H, AA'BB' system, ^3J = 8.5, H Ar); 7.86 (2H, AA'BB' system, ^3J = 8.5, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.6; 38.9; 106.8; 109.3; 128.6; 130.3 (2C); 132.1 (2C); 135.4; 146.1; 152.0; 194.3.

1-(4-Bromophenyl)-2-(5-*tert*-butylfuran-2-yl)ethanone (5b). Yield 1.86 g (58%), light-beige powder, mp 57–60°C (4:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 1689 (C=O), 1584, 1334, 1209, 1071, 994. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.23 (9H, s, $C(CH_3)_3$); 4.21 (2H, s, CH_2); 5.87 (1H, d, ^3J = 3.1, H Fur); 6.07 (1H, d, ^3J = 3.1, H Fur); 7.60 (2H, AA'BB' system, ^3J = 8.6, H Ar); 7.87 (2H, AA'BB' system, ^3J = 8.6, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 29.2 (3C); 32.7; 39.1; 103.1; 108.7; 128.5; 130.4 (2C); 132.0 (2C); 135.3; 145.6; 164.1; 194.4. Mass spectrum (EI), *m*/*z* (I_{rel}, %): 322 $[M(^{81}Br)]^+$ (2), 320 $[M(^{81}Br)]^+$ (2), 185 (14), 183 (15), 159 (5), 157 (5), 137 (100), 122 (9), 107 (9), 95 (7), 76 (5). Found, %: C 59.72; H 5.30. $C_{16}H_{17}BrO_2$. Calculated, %: C 59.83; H 5.33.

1-(4-Bromophenyl)-2-[5-(4-chlorophenyl)furan-2-yl]-ethanone (5c). Yield 2.06 g (55%), light-beige powder, mp 118–120°C (2:1 petroleum ether–CH_2Cl_2). IR spectrum (Nujol), ν, cm^{-1}: 1689 (C=O), 1582, 1221, 1096, 1074, 1016, 960. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 4.33 (2H, s, CH_2); 6.32 (1H, d, ^3J = 3.3, H Fur); 6.58 (1H, d, ^3J = 3.3, H Fur); 7.32 (2H, AA'BB' system, ^3J = 8.6, H Ar); 7.53 (2H, AA'BB' system, ^3J = 8.6, H Ar); 7.63 (2H, AA'BB' system, ^3J = 8.0, H Ar); 7.90 (2H, AA'BB' system, ^3J = 8.0, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 38.8; 106.7; 110.9; 125.0 (2C); 128.9; 129.0 (2C); 129.3; 130.3 (2C); 132.2 (2C); 133.0; 135.0; 148.0; 152.8; 193.8. Mass spectrum (EI), *m*/*z* (I_{rel}, %): 378/377/376/375/374 $[M]^+$ (3/2/10/1/6), 193 (35), 191 (100), 157 (10), 155 (10), 128 (25), 76 (10). Found, %: C 57.61; H 3.27. $C_{18}H_{12}BrClO_2$. Calculated, %: C 57.55; H 3.22.

1-(4-Bromophenyl)-2-(furan-2-yl)ethanone (5d). Yield 1.40 g (53%), light-yellow powder, mp 97–98°C (9:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 1698 (C=O), 1586, 1465, 1328, 1071, 993. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 4.27 (2H, s, CH_2); 6.23 (1H, d, ^3J = 3.0, H Fur); 6.34 (1H, dd, ^3J = 3.0, ^4J =2.0, H Fur); 7.37 (1H, d, ^4J = 2.0, H Fur); 7.61 (2H, AA'BB' system, ^3J = 8.5, H Ar); 7.87 (2H, AA'BB' system, ^3J = 8.5, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 38.7; 108.6; 110.9; 128.8; 130.3 (2C); 132.2 (2C); 135.1; 142.4; 148.0; 194.2. Mass spectrum (EI), *m*/*z* (I_{rel}, %): 266 $[M(^{81}Br)]^+$ (8), 264 $[M(^{79}Br)]^+$ (8), 185 (98), 183 (100), 157 (34), 155 (35), 104 (15), 76 (40), 53 (30). Found, %: C 54.41; H 3.37. $C_{12}H_9BrO_2$. Calculated, %: C 54.37; H 3.42.

2-(5-Methylfuran-2-yl)-1-(4-nitrophenyl)ethanone (5e).[25] Yield 1.35 g (55%), light-beige needles, mp 67–69°C (4:1 petroleum ether–EtOAc) (mp 67–69°C (petroleum ether–EtOAc)[25]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.24 (3H, s, CH_3); 4.28 (2H, s, CH_2); 5.91 (1H, d, ^3J = 3.0, H Fur); 6.11 (1H, d, ^3J = 3.0, H Fur); 8.15 (2H, AA'BB' system, ^3J = 8.5, H Ar); 8.29 (2H, AA'BB' system, ^3J = 8.5, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.6; 39.3; 106.8; 109.7; 124.0 (2C); 129.8 (2C); 140.8; 145.1; 150.5; 152.3; 193.9.

1-(4-Methoxyphenyl)-2-(5-methylfuran-2-yl)ethanone (5f).[25] Yield 1.33 g (58%), light-beige powder, mp 69–70°C (4:1 petroleum ether–diethyl ether) (mp 69–70°C (petroleum ether–EtOAc)[25]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.25 (3H, s, CH_3); 3.86 (3H, s, OCH_3); 4.20 (2H, s, CH_2); 5.89 (1H, d, ^3J = 3.0, H Fur); 6.07 (1H, d, ^3J = 3.0, H Fur); 6.93 (2H, AA'BB' system, ^3J = 8.8, H Ar); 7.99 (2H, AA'BB' system, ^3J = 8.8, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.7; 38.5; 55.6; 106.6; 108.9; 113.9 (2C); 129.4; 131.1 (2C); 146.8; 151.7; 163.8; 194.1.

1-(3,4-Dimethoxyphenyl)-2-(5-methylfuran-2-yl)etha-none (5g). Yield 1.66 g (64%), colorless needles, mp 116–

117°C (4:1 petroleum ether–EtOAc). IR spectrum (KBr), ν, cm^{-1}: 1677 (C=O), 1582, 1517, 1417, 1265, 1162, 1019, 795. ^{1}H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.24 (3H, s, CH_3); 3.91 (3H, s, OCH_3); 3.93 (3H, s, OCH_3); 4.19 (2H, s, CH_2); 5.89 (1H, d, ^3J = 3.0, H Fur); 6.07 (1H, d, ^3J = 3.0, H Fur); 6.88 (1H, d, ^3J = 8.4, H Ar); 7.56 (1H, d, ^4J = 2.0, H Ar); 7.65 (1H, dd, ^3J = 8.4, ^4J = 2.0, H Ar). ^{13}C NMR spectrum (75 MHz, $CDCl_3$), δ, ppm: 13.6; 38.4; 56.0; 56.2; 106.6; 108.9; 110.1; 110.8; 123.6; 129.6; 146.8; 149.1; 151.6; 153.5; 194.0. Mass spectrum (EI), *m/z* (I_{rel}, %): 260 $[M]^+$ (25), 165 (100), 137 (33), 122 (32), 107 (24), 95 (70), 77 (66), 43 (38). Found, %: C 69.13; H 6.29. $C_{15}H_{16}O_4$. Calculated, %: C 69.22; H 6.20.

1-(4,5-Dimethoxy-2-nitrophenyl)-2-(5-methylfuran-2-yl)-ethanone (5h). Yield 1.59 g (52%), light-yellow needles, mp 117–118°C (3:1 petroleum ether–CH_2Cl_2). IR spectrum (KBr), ν, cm^{-1}: 1706 (C=O), 1578, 1524, 1334, 1287, 1229, 1176, 1084, 1024, 950. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.19 (3H, s, CH_3); 3.90 (3H, s, OCH_3); 3.96 (3H, s, OCH_3); 4.05 (2H, s, CH_2); 5.85 (1H, d, ^3J = 3.0, H Fur); 6.03 (1H, d, ^3J = 3.0, H Fur); 6.59 (1H, s, H Ar); 7.60 (1H, s, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.6; 42.7; 56.6; 56.7; 106.7; 106.8; 109.3; 110.0; 132.0; 138.3; 145.6; 149.6; 152.1; 154.0; 198.7. Mass spectrum (EI), *m/z* (I_{rel}, %): 305 $[M]^+$ (1), 211 (4), 181 (3), 136 (7), 121 (3), 110 (10), 95 (100). Found, %: C 58.91; H 5.03; N 4.67. $C_{15}H_{15}NO_6$. Calculated, %: C 59.01; H 4.95; N 4.59.

1-(4-Bromophenyl)-2-(methylsulfonyl)ethanone (6a). Isolated along with the product **5a**. Yield 0.61 g (22%), light-yellow needles, mp 142–144°C (EtOAc) (mp 134–139°C[35]). ^{1}H NMR spectrum (400 MHz), δ, ppm (*J*, Hz): 3.13 (3H, s, CH_3); 4.55 (2H, s, CH_2); 7.68 (2H, AA'BB' system, ^3J = 8.6, H Ar); 7.87 (2H, AA'BB' system, ^3J = 8.6, H Ar). ^{13}C NMR spectrum (100 MHz), δ, ppm: 42.0; 61.6; 130.6; 130.9 (2C); 132.6 (2C); 134.6; 188.5.

1,4-Bis(4-bromophenyl)butane-1,4-dione (7a). Isolated along with the product **5a**. Yield 0.28 g (7%), light-beige needles, mp 174–175°C (ethanol) (mp 176–178°C[36]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 3.40 (4H, s, CH_2); 7.60 (4H, AA'BB' system, ^3J = 8.3, H Ar); 7.87 (4H, AA'BB' system, ^3J = 8.3, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 32.9; 128.8; 130.1; 132.4; 135.8; 197.9.

2-(Methylsulfonyl)-1-(4-nitrophenyl)ethanone (6e). Isolated along with the product **5e**. Yield 0.75 g (31%), light-yellow needles, mp 155–157°C (EtOAc) (mp 148.9–149.4°C (hexane–CH_2Cl_2)[37]). ^{1}H NMR spectrum (600 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 3.16 (3H, s, CH_3), 4.65 (2H, s, CH_2); 8.19 (2H, AA'BB' system, ^3J = 8.8, H Ar); 8.37 (2H, AA'BB' system, ^3J = 8.8, H Ar). ^{13}C NMR spectrum (150 MHz, $CDCl_3$), δ, ppm: 41.9; 61.9; 124.3 (2C); 130.6 (2C); 140.0; 151.2; 188.2.

1,4-Bis(4-nitrophenyl)butane-1,4-dione (7e). Isolated along with the product **5e**. Yield 0.16 g (5%), yellow needles, mp 178–180°C (ethanol) (mp 195–196°C ($CHCl_3$)[38]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 3.51 (4H, s, CH_2); 8.17 (4H, AA'BB' system, ^3J = 7.3, H Ar); 8.33 (4H, AA'BB' system, ^3J = 8.3, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 33.4; 124.4; 129.6; 141.3; 150.9; 197.2.

1-(3,4-Dimethoxyphenyl)-2-(methylsulfonyl)ethanone (6g). Isolated along with the product **5g**. Yield 0.65 g (25%), white powder, mp 142–143°C (EtOAc). IR spectrum (KBr), ν, cm^{-1}: 1675 (C=O), 1516, 1303, 1152, 1119, 1020, 976. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 3.11 (3H, s, CH_3); 3.91 (3H, s, OCH_3); 3.93 (3H, s, OCH_3); 4.55 (2H, s, CH_2); 6.90 (1H, d, ^3J = 8.5, H Ar); 7.50 (1H, d, ^4J = 2.0, H Ar); 7.60 (1H, dd, ^3J = 8.5, ^4J = 2.0, H Ar). ^{13}C NMR spectrum (150 MHz, $CDCl_3$), δ, ppm: 41.7; 56.0; 56.2; 61.1; 110.3; 110.6; 125.0; 128.8; 149.4; 154.8; 187.4. Mass spectrum (EI), *m/z* (I_{rel}, %): 258 $[M]^+$ (100), 166 (22), 165 (90), 151 (13), 137 (14), 43 (20). Found, %: C 51.22; H 5.51. $C_{11}H_{14}O_5S$. Calculated, %: C 51.15; H 5.46.

1,4-Bis(3,4-dimethoxyphenyl)butane-1,4-dione (7g).[27] Isolated along with the product **5g**. Yield 0.18 g (5%), colorless needles, mp 185–187°C (ethanol) (mp 176–178°C[27]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 3.41 (4H, s, CH_2); 3.92 (6H, s, OCH_3); 6.89 (2H, d, ^3J = 8.1, H Ar); 7.55 (2H, br. s, H Ar); 6.89 (2H, br. d, ^3J = 8.1, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 32.7; 56.4; 56.5; 110.5; 110.6; 123.2; 130.5; 149.4; 153.7; 197.9.

2-Chloroacetamides 10a–i were obtained according to the described procedure and used in further reactions without identification.[39]

Preparation of xanthogenates 11a–i (General method). A solution of 2-chloroacetamide **10a–i** (18 mmol) in anhydrous acetone (60 ml) was cooled (5–10°C) and treated by portionwise addition of potassium ethyl xanthogenate (3.52 g, 22 mmol). The reaction mixture was stirred for 30 min at room temperature, then refluxed for 3 h. The solvent was evaporated to dryness at reduced pressure. The obtained residue in flask was treated with 200 ml of cold water, the precipitate that formed was filtered off, washed with water (3×50 ml), and air-dried.

***S*-(2-Amino-2-oxoethyl) *O*-ethyl dithiocarbonate (11a)**. Yield 2.51 g (78%), white powder, mp 114–115°C (4:1 petroleum ether–EtOAc) (mp 114–115°C[40]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.43 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 3.85 (2H, s, CH_2); 4.67 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 6.05 (1H, br. s, NH); 6.27 (1H, br. s, NH). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.8; 38.9; 71.3; 169.8; 213.0.

***S*-[2-(Benzylamino)-2-oxoethyl)] *O*-ethyl dithiocarbonate (11b)**. Yield 3.87 g (80%), white powder, mp 86–88° C (4:1 petroleum ether–EtOAc) (mp 94–95°C[41]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.42 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 3.92 (2H, s, CH_2); 4.48 (2H, d, ^3J = 5.8, $C\underline{H}_2NH$); 4.66 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 6.66 (1H, br. s, NH); 7.28–7.37 (5H, m, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.8; 39.3; 44.0; 71.3; 127.7; 127.8 (2C); 128.8 (2C); 137.9; 167.0; 213.1.

***O*-Ethyl *S*-{2-[(4-fluorophenyl)amino]-2-oxoethyl} dithiocarbonate (11c)**. Yield 4.37 g (89%), light-beige needles, mp 108–109°C (4:1 petroleum ether–EtOAc). IR spectrum (KBr), ν, cm^{-1}: 3306 (N–H), 1667 (C=O), 1549, 1362, 1229, 1113, 1047. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.43 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$);

3.99 (2H, s, CH_2); 4.68 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 6.97–7.03 (2H, m, H Ar); 7.42–7.47 (2H, m, H Ar); 8.28 (1H, br. s, NH). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 13.9; 40.0; 71.7; 115.8 (2C, d, $^2J_{CF}$ = 22.5); 122.1 (2C, d, $^3J_{CF}$ = 7.9); 133.5 (1C, d, $^4J_{CF}$ = 2.8); 159.7 (1C, d, $^1J_{CF}$ = 244.2); 165.6; 213.9. Mass spectrum (EI), *m/z* (I_{rel}, %): 273 $[M]^+$ (48), 184 (19), 163 (98), 150 (18), 135 (21), 124 (27), 111 (100), 95 (27), 82 (16), 47 (43), 43 (35). Found, %: C 48.27; H 4.39; N 5.16. $C_{11}H_{12}FNO_2S_2$. Calculated, %: C 48.33; H 4.42; N 5.12.

***O*-Ethyl *S*-{2-[(4-nitrophenyl)amino]-2-oxoethyl} dithiocarbonate (11d)**. Yield 4.75 g (88%), light-beige needles, mp 108–109°C (4:1 petroleum ether–EtOAc). IR spectrum (ATR), ν, cm^{-1}: 3360 (NH), 1680 (C=O), 1552, 1493, 1333, 1252, 1114, 1051, 851. ^{1}H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.45 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 4.05 (2H, s, CH_2); 4.70 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 7.69 (2H, AA'BB' system, ^3J = 9.1, H Ar); 8.20 (2H, AA'BB' system, ^3J = 9.1, H Ar); 8.69 (1H, br. s, NH). ^{13}C NMR spectrum (75 MHz, $CDCl_3$), δ, ppm: 13.9; 40.3; 72.1; 119.5 (2C); 125.2 (2C); 143.4; 143.9; 166.3; 214.3. Mass spectrum (EI), *m/z* (I_{rel}, %): 300 $[M]^+$ (100), 277 (13), 240 (54), 211 (52), 180 (31), 163 (84), 147 (23), 138 (56), 122 (23), 108 (27), 95 (21), 75 (26), 43 (42). Found, %: C 44.07; H 4.11; N 9.38. $C_{11}H_{12}N_2O_4S_2$. Calculated, %: C 43.99; H 4.03; N 9.33.

***O*-Ethyl *S*-{2-[(2-nitrophenyl)amino]-2-oxoethyl} dithiocarbonate (11e)**. Yield 4.43 g (82%), light-yellow needles, mp 89–91°C (4:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 3306 (NH), 1664 (C=O), 1552, 1511, 1230, 1114, 1050. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.44 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 4.10 (2H, s, CH_2); 4.71 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 7.17–7.23 (1H, m, H Ar); 7.63–7.67 (1H, m, H Ar); 8.19–8.21 (1H, m, H Ar); 8.72–8.74 (1H, m, H Ar); 10.90 (1H, br. s, NH). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.9; 40.9; 71.5; 122.6; 123.9; 125.9; 134.3; 135.9; 137.1; 166.4; 212.1. Mass spectrum (ESI), *m/z* (I_{rel}, %): 323 $[M+Na]^+$ (100). Found, %: C 44.04; H 4.09; N 9.36. $C_{11}H_{12}N_2O_4S_2$. Calculated, %: C 43.99; H 4.03; N 9.33.

***O*-Ethyl *S*-[2-oxo-2-(pyridin-2-ylamino)ethyl] dithiocarbonate (11f)**. Yield 3.59 g (78%), white powder, mp 98–100°C (4:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 1667 (C=O), 1577, 1551, 1433, 1328, 1287, 1229, 1205, 1177, 1117, 1054. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.44 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 4.04 (2H, s, CH_2); 4.70 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 7.03–7.06 (1H, m, H Py); 7.68–7.73 (1H, m, H Py); 8.17–8.19 (1H, m, H Py); 8.26–8.28 (1H, m, H Py); 8.71 (1H, br. s, NH). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.9; 40.5; 71.5; 114.3; 120.3; 138.6; 147.9; 151.2; 165.8; 212.9. Mass spectrum (EI), *m/z* (I_{rel}, %): 256 $[M]^+$ (3), 210 (25), 168 (30), 137 (28), 78 (100), 59 (16). Found, %: C 46.94; H 4.69; N 10.86. $C_{10}H_{12}N_2O_2S_2$. Calculated, %: C 46.85; H 4.72; N 10.93.

***O*-Ethyl *S*-(2-{[5-ethyl-3-(ethoxycarbonyl)thiophen-2-yl]amino}-2-oxoethyl) dithiocarbonate (11g)**. Yield 5.46 g (84%), white powder, mp 48–50°C (9:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 3285 (N–H), 1677 (C=O), 1562, 1535, 1407, 1295, 1241, 1204, 1179, 1114, 1050. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.29 (3H, t, ^3J = 7.5, $CH_2C\underline{H}_3$); 1.37 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 1.42 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 2.74 (2H, q, ^3J = 7.5, $C\underline{H}_2CH_3$); 4.07 (2H, s, CH_2); 4.34 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 4.69 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 6.88 (1H, s, H Th); 11.41 (1H, br. s, NH). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.8; 14.5; 15.7; 23.0; 39.4; 60.8; 71.2; 113.4; 119.5; 137.7; 146.1; 164.3; 165.3; 211.8. Mass spectrum (ESI), *m/z* (I_{rel}, %): 384 $[M+Na]^+$ (99), 163 (100). Found, %: C 46.64; H 5.19; N 3.86. $C_{14}H_{19}NO_4S_3$. Calculated, %: C 46.51; H 5.30; N 3.87.

(4-Methoxyphenyl) 2-[(ethoxycarbothioyl)sulfanyl]acetate (11h). Yield 4.53 g (88%), light-yellow oil. IR spectrum (Nujol), ν, cm^{-1}: 1762 (C=O), 1596, 1504, 1235, 1192, 1109, 1051, 938. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.44 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 3.79 (3H, s, OCH_3); 4.12 (2H, s, CH_2); 4.68 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 6.88 (2H, AA'BB' system, ^3J = 9.1, H Ar); 7.03 (2H, AA'BB' system, ^3J = 9.1, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.9; 38.0; 55.7; 71.0; 114.6 (2C); 122.2 (2C); 144.3; 157.6; 167.0; 212.6. Mass spectrum (EI), *m/z* (I_{rel}, %): 286 $[M]^+$ (1), 163 (100), 135 (84), 124 (68), 109 (60), 95 (19), 81 (15), 65 (10). Found, %: C 50.28; H 4.99. $C_{12}H_{14}O_4S_2$. Calculated, %: C 50.33; H 4.93.

(4-Fluorophenyl) 2-[(ethoxycarbothioyl)sulfanyl]acetate (11i). Yield 4.39 g (89%), colorless needles, mp 43–45°C (3:1 petroleum ether–CH_2Cl_2). IR spectrum (Nujol), ν, cm^{-1}: 1760 (C=O), 1502, 1308, 1229, 1192, 1150, 1110, 1046, 946. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.44 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 4.13 (2H, s, CH_2); 4.68 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 7.04–7.11 (4H, m, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 13.2; 37.3; 70.4; 115.6 (2C, d, $^2J_{CF}$ = 23.5); 122.2 (2C, d, $^3J_{CF}$ = 8.7); 145.9 (1C, d, $^4J_{CF}$ = 2.9); 159.8 (1C, d, $^1J_{CF}$ = 245.0); 166.1; 211.9. Mass spectrum (EI), *m/z* (I_{rel}, %): 163 $[M–4\text{-}FC_6H_4O]^+$ (100), 135 (93), 112 (78), 107 (43), 93 (11), 83 (37), 75 (11), 57 (16). Found, %: C 48.21; H 4.00. $C_{11}H_{11}FO_3S_2$. Calculated, %: C 48.16; H 4.04.

2-(5-Methylfuran-2-yl)acetamide (12a). Yield 0.86 g (62%), beige powder, mp 124–126°C (4:1 petroleum ether–EtOAc) (mp 112–114°C (petroleum ether)[42]). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.25 (3H, s, CH_3); 3.53 (2H, s, CH_2); 5.79 (1H, br. s, NH); 5.90 (1H, d, ^3J = 2.8, H Fur); 6.08 (1H, d, ^3J = 2.8, H Fur); 6.16 (1H, br. s, NH). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.6; 36.1; 106.7; 109.4; 146.9; 152.3; 172.0.

***N*-Benzyl-2-(5-methylfuran-2-yl)acetamide (12b)**. Yield 1.44 g (63%), white powder, mp 65–67°C (4:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 3286 (NH), 1644 (C=O), 1545, 1262, 1160, 1080, 1016. ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.25 (3H, s, CH_3); 3.60 (2H, s, CH_2); 4.45 (2H, d, ^3J = 5.8, $C\underline{H}_2NH$); 5.90 (1H, d, ^3J = 2.8, H Fur); 6.03 (1H, br. s, NH); 6.10 (1H, d, ^3J = 2.8, H Fur); 7.22–7.34 (5H, m, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.6; 36.6; 43.7; 106.8; 109.6; 127.5; 127.6 (2C); 128.8 (2C); 138.3; 146.9; 152.4; 169.0. Mass spectrum (EI), *m/z* (I_{rel}, %): 229 $[M]^+$

(12), 147 (5), 96 (68), 95 (100), 91 (40), 65 (11). Found, %: C 73.42; H 6.58; N 6.14. $C_{14}H_{15}NO_2$. Calculated, %: C 73.34; H 6.59; N 6.11.

***N*-(4-Fluorophenyl)-2-(5-methylfuran-2-yl)acetamide (12c)**. Yield 1.56 g (67%), colorless needles, mp 116–117°C (4:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 3276 (N–H), 1658 (C=O), 1533, 1507, 1218, 1032, 833. ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.29 (3H, s, CH_3); 3.69 (2H, s, CH_2); 5.96 (1H, d, ^3J = 3.0, H Fur); 6.17 (1H, d, ^3J = 3.0, H Fur); 6.95–7.00 (2H, m, H Ar); 7.40–7.44 (2H, m, H Ar); 7.66 (1H, br. s, NH). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 13.7; 37.3; 106.9; 110.0; 115.7 (2C, d, $^2J_{CF}$ = 22.5); 122.0 (2C, d, ^3J = 7.9); 133.7 (1C, d, $^4J_{CF}$ = 2.9); 146.4; 152.7; 159.6 (1C, d, $^1J_{CF}$ = 243.7); 167.3. Mass spectrum (EI), *m/z* (I_{rel}, %): 233 $[M]^+$ (94), 150 (10), 138 (25), 122 (38), 111 (45), 96 (66), 95 (100), 81 (36), 67 (22), 43 (32). Found, %: C 66.92; H 5.23; N 5.96. $C_{13}H_{12}FNO_2$. Calculated, %: C 66.94; H 5.19; N 6.01.

2-(5-Methylfuran-2-yl)-*N*-(4-nitrophenyl)acetamide (12d). Yield 1.96 g (65%), light-beige powder, mp 138–139°C (4:1 petroleum ether–EtOAc). IR spectrum (ATR), ν, cm^{-1}: 3266 (N–H), 1665 (C=O), 1552, 1512, 1342, 1256, 854. ^1H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.30 (3H, s, CH_3); 3.76 (2H, s, CH_2); 5.98 (1H, d, ^3J = 3.0, H Fur); 6.21 (1H, d, ^3J = 3.0, H Fur); 7.67 (2H, AA'BB' system, ^3J = 9.1, H Ar); 7.93 (1H, br. s, NH); 8.18 (2H, AA'BB' system, ^3J = 9.1, H Ar). ^{13}C NMR spectrum (75 MHz, $CDCl_3$), δ, ppm: 13.7; 37.7; 107.1; 110.4; 119.3 (2C); 125.1 (2C); 143.5; 143.7; 145.6; 153.1; 167.6. Mass spectrum (EI), *m/z* (I_{rel}, %): 260 $[M]^+$ (54), 165 (11), 150 (26), 122 (15), 104 (12), 96 (100), 95 (98), 81 (24), 76 (16), 59 (20), 53 (30), 44 (54). Found, %: C 59.94; H 4.61; N 10.82. $C_{13}H_{12}N_2O_4$. Calculated, %: C 60.00; H 4.65; N 10.76.

2-(5-Methylfuran-2-yl)-*N*-(2-nitrophenyl)acetamide (12e). Yield 1.61 g (62%), light-yellow powder, mp 65–67°C (4:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 3263 (N–H), 1675 (C=O), 1592, 1574, 1532, 1521, 1365, 1314, 1215, 1196, 1023. ^1H NMR spectrum (400 MHz, DMSO-d_6), δ, ppm (*J*, Hz): 2.24 (3H, s, CH_3); 3.60 (2H, s, CH_2); 6.02 (1H, d, ^3J = 3.0, H Fur); 6.20 (1H, d, ^3J = 3.0, H Fur); 7.32–7.36 (1H, m, H Ar); 7.68–7.72 (1H, m, H Ar); 7.90–7.92 (1H, m, H Ar); 7.98–8.00 (1H, m, H Ar); 10.34 (1H, br. s, NH). ^{13}C NMR spectrum (100 MHz, DMSO-d_6), δ, ppm: 13.2; 36.0; 106.6; 109.0; 124.5; 124.9; 125.0; 131.6; 134.3; 141.2; 146.5; 151.0; 167.3. Mass spectrum (EI), *m/z* (I_{rel}, %): 260 $[M]^+$ (14), 122 (15), 95 (100), 79 (5), 65 (6), 55 (5).

2-(5-Methylfuran-2-yl)-*N*-(pyridin-2-yl)acetamide (12f). Yield 1.34 g (62%). Light-beige powder, mp 94–95°C (4:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 3239 (N–H), 1666 (C=O), 1578, 1537, 1434, 1300, 1221, 775. ^1H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.28 (3H, s, CH_3); 3.75 (2H, s, CH_2); 5.94 (1H, d, ^3J = 3.0, H Fur); 6.19 (1H, d, ^3J = 3.0, H Fur); 7.02–7.07 (1H, m, H Py); 7.69–7.75 (1H, m, H Py); 8.23–8.26 (2H, m, H Py); 8.63 (1H, br. s, NH). ^{13}C NMR spectrum (75 MHz, $CDCl_3$), δ, ppm: 13.7; 37.7; 106.9; 110.2; 114.4; 120.0; 139.1; 145.7; 147.1; 151.1; 152.8; 167.8. Mass spectrum (EI), *m/z* (I_{rel}, %): 216 $[M]^+$ (90), 122 (42), 96 (20), 95 (100), 79 (21), 78 (74), 67 (22), 51 (24), 43 (32). Found, %: C 66.71; H 5.63; N 12.99. $C_{12}H_{12}N_2O_2$. Calculated, %: C 66.65; H 5.59; N 12.96.

Ethyl 5-ethyl-2-[2-(5-methylfuran-2-yl)acetamido]-thiophene-3-carboxylate (12g). Yield 1.73 g (54%), light-yellow prisms, mp 77–78°C (9:1 petroleum ether–EtOAc). IR spectrum (Nujol), ν, cm^{-1}: 3253 (N–H), 1661 (C=O), 1530, 1401, 1226, 1037, 782. ^1H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 1.27 (3H, t, ^3J = 7.5, $CH_2C\underline{H}_3$); 1.33 (3H, t, ^3J = 7.1, $OCH_2C\underline{H}_3$); 2.33 (3H, s, CH_3); 2.71 (2H, q, ^3J = 7.5, $C\underline{H}_2CH_3$); 3.80 (2H, s, CH_2); 4.27 (2H, q, ^3J = 7.1, $OC\underline{H}_2CH_3$); 5.97 (1H, d, ^3J = 3.0, H Fur); 6.20 (1H, d, ^3J = 3.0, H Fur); 6.83 (1H, s, H Th); 11.11 (1H, br. s, NH). ^{13}C NMR spectrum (75 MHz, $CDCl_3$), δ, ppm: 13.6; 14.5; 15.7; 22.9; 36.5; 60.5; 106.7; 110.0; 112.8; 119.2; 137.1; 145.2; 146.4; 153.0; 165.2; 166.1. Mass spectrum (EI), *m/z* (I_{rel}, %): 321 $[M]^+$ (61), 226 (22), 216 (18), 199 (100), 184 (40), 180 (38), 153 (46), 138 (31), 122 (39), 96 (42), 95 (45), 58 (37), 44 (52), 42 (41). Found, %: C 59.68; H 6.01; N 4.33. $C_{16}H_{19}NO_4S$. Calculated, %: C 59.79; H 5.96; N 4.36.

(4-Methoxyphenyl) 2-(5-methylfuran-2-yl)acetate (12h). Yield 1.33 g (54%), light-yellow oil. IR spectrum (Nujol), ν, cm^{-1}: 1762 (C=O), 1597, 1571, 1506, 1341, 1298, 1249, 1192, 1124, 1022, 971. ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.29 (3H, s, CH_3); 3.79 (3H, s, OCH_3); 3.85 (2H, s, CH_2); 5.94 (1H, d, ^3J = 3.0, H Fur); 6.18 (1H, d, ^3J = 3.0, H Fur); 6.88 (2H, AA'BB' system, ^3J = 9.0, H Ar); 7.02 (2H, AA'BB' system, ^3J = 9.0, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm: 13.7; 34.4; 55.7; 106.6; 109.2; 114.6 (2C); 122.3 (2C); 144.4; 145.3; 152.0; 157.5; 168.6. Mass spectrum (EI), *m/z* (I_{rel}, %): 246 $[M]^+$ (6), 122 (69), 109 (8), 95 (100), 79 (5). Found, %: C 68.23; H 6.01. $C_{14}H_{14}O_4$. Calculated, %: C 68.28; H 5.73.

(4-Fluorophenyl) 2-(5-methylfuran-2-yl)acetate (12i). Yield 1.31 g (56%), light-yellow oil. IR spectrum (Nujol), ν, cm^{-1}: 1764 (C=O), 1569, 1503, 1341, 1186, 1124, 1022, 971. ^1H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.29 (3H, s, CH_3); 3.86 (2H, s, CH_2); 5.94 (1H, d, ^3J = 2.9, H Fur); 6.18 (1H, d, ^3J = 2.9, H Fur); 7.02–7.26 (4H, m, H Ar). ^{13}C NMR spectrum (100 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 13.6; 34.3; 106.6; 109.3; 116.2 (2C, d, $^2J_{CF}$ = 23.6); 123.0 (2C, d, $^3J_{CF}$ = 8.6); 145.0; 146.7 (1C, d, $^4J_{CF}$ = 3.0); 152.2; 160.4 (1C, d, $^1J_{CF}$ = 244.3); 168.3. Mass spectrum (EI), *m/z* (I_{rel}, %): 234 $[M]^+$ (7), 122 (20), 112 (3), 95 (100), 83 (6). Found, %: C 66.79; H 4.81. $C_{13}H_{11}FO_3$. Calculated, %: C 66.66; H 4.73.

The Supplementary file to this article containing ^1H and ^{13}C NMR spectra of the synthesized compounds is available for authorized users.

The results of this work were obtained with financial support from the Grants Council of the President of the Russian Federation (*grant MK-1476.2014.3) and the Ministry of Education and Science of the Russian Federation (State Orders No. 2754 and No. 4.246.2014/K).*

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