CYCLOHEXYLIDENEACETYL ISOTHIOCYANATE. PRECURSOR FOR THE SYNTHESIS OF 1,3-THIAZASPIRO-CYCLOALKANES AND 1,3-DIAZASPIROCYCLOALKANES

Marcel TOROK[a], Milan DZURILLA[a], Peter KUTSCHY[a], Jan IMRICH[a], Ivan DANIHEL[a], Paul RADEMACHER[b] and Heinz BANDMANN[b]

[a] *Department of Organic Chemistry, Safarik University, 04001 Kosice, The Slovak Republic*
[b] *Institute of Organic Chemistry, University of Essen, D-45117 Essen, Germany*

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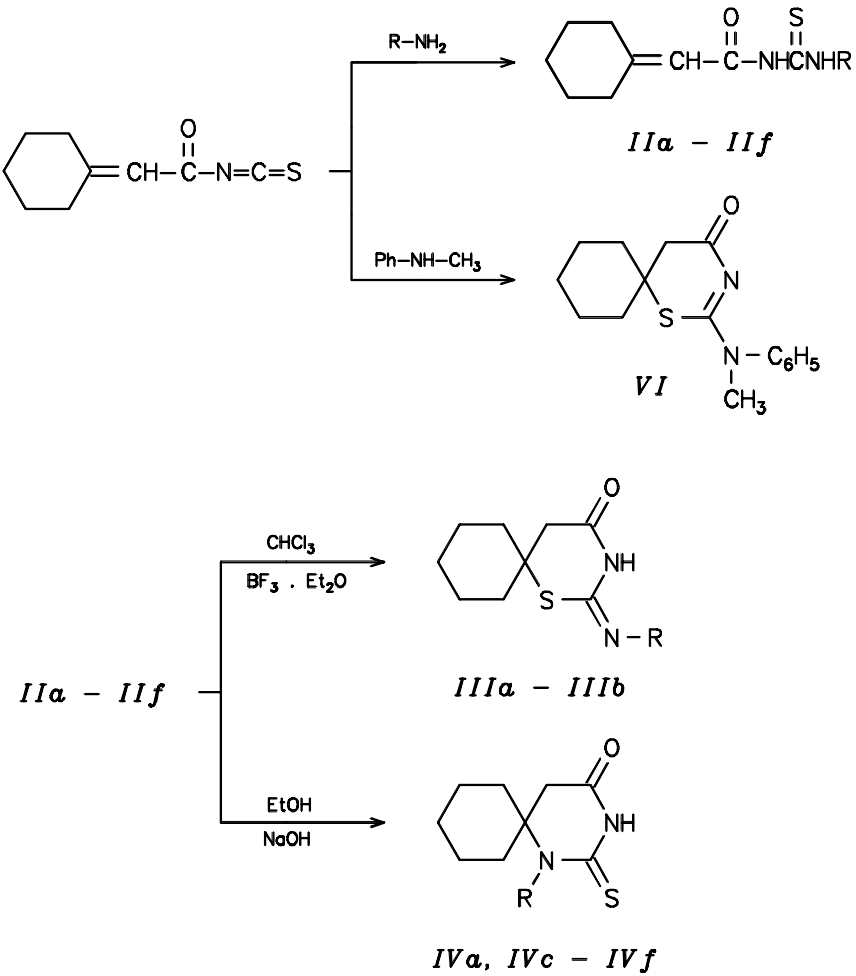
The *N*-(4-substituted phenyl)-*N'*-cyclohexylideneacetylthioureas *IIa – IIf* were readily prepared by the reaction of cyclohexylideneacetyl isothiocyanate *I* with 4-substituted anilines. They were cyclized to the 2-(4-substituted phenylimino)-4-oxo-1,3-thiazaspiro[5.5]undecanes *IIIa – IIIe* in the presence of boron trifluoride etherate and to the 1-(4-substituted phenyl)-4-oxo-2-thioxo-1,3-diazaspiro[5.5]-undecanes *IVa, IVc – IVf* in alkaline medium.

Isothiocyanates are used for a long time as convenient starting compounds for the synthesis of heterocycles containing the N–C–S grouping[1]. Among them the interesting compounds are α,β-unsaturated acyl isothiocyanates. The presence of C=C and C=O groups in the molecules of these compounds significantly contributes to their high synthetic potential in the synthesis of thiazolines[2], pyrimidines[3] and 1,3-thiazines[4].

We started the synthesis of cyclohexylideneacetyl isothiocyanate *I* from cyclohexylideneacetic acid which was transformed to the corresponding acid chloride by the action of thionyl chloride, then treatment with lead(II) thiocyanate afforded isothiocyanate *I*.

Isothiocyanate *I* reacts with primary amines in acetone at room temperature to give the *N*-(4-substituted phenyl)-*N'*-2-cyclohexylideneacetylthioureas[5]. In the case of *N*-methylaniline it is not possible to isolate the corresponding thiourea and instead the cyclization product *VI* is obtained (Scheme 1). The presence of the –CS–NH–R grouping and an electrophilic centre on the β-carbon atom of the C=C double bond enables selective intramolecular cyclization via the nitrogen or sulfur atom. It is known in the literature that in basic medium the *N*-cyclization is preferred[6] whereas by heating[7] or under Lewis acid catalysis[4] 1,3-thiazines are formed.

The complexation of boron trifluoride with the carbonyl oxygen of thioureas *IIa – IIe* decreases significantly the energy of the lowest unoccupied molecular orbital (LUMO) and increases the orbital coefficient on the β-carbon of the Michael acceptor[8]. Consequently, the intramolecular cyclization via the more nucleophilic sulfur atom with for-



In formulae *II – IV* : *a*, R = C_6H_5 *d*, R = 4-CH_3-C_6H_4
b, R = 4-NO_2-C_6H_4 *e*, R = 4-CH_3O-C_6H_4
c, R = 4-Br-C_6H_4 *f*, R = 4-CH_3CO-C_6H_4

SCHEME 1

mation of 2-(4-substituted phenylimino)-4-oxo-1,3-thiazaspiro[5.5]undecanes *IIIa – IIIe* is preferred (Scheme 1). This type of 1,3-thiazines can exist in the amino as well as the imino form. We have studied this tautomerism by IR spectroscopy and ^{13}C NMR spectroscopy. The IR spectra taken in $CHCl_3$ and KBr have been compared with the spectra of compound *VI* with the endocyclic C=N bond and the results for 6-phenyl-2-thioxo-2,3-dihydro-4*H*-1,3-thiazin-4-one (ref.[9]) are in accordance with our assumption showing only the absorption bands of the imino form (Table I). As a result of conjugation in thiazine *VI* the bands of the C=O and C=N groups are shifted to lower wavenumbers by about 30 and 80 cm^{-1}, respectively. Important differences between *III* and *VI* were observed in the ^{13}C NMR spectra. The amino form exhibits a downfield shift of the carbonyl carbon atom (~10 ppm) and the carbon atom in position 2 (~15 ppm) (Table I).

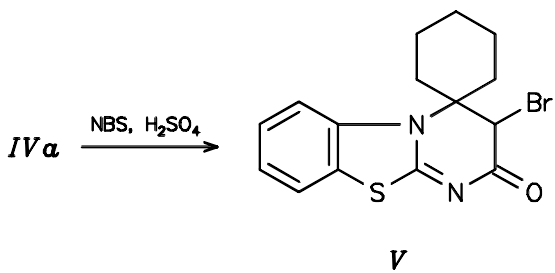
Cyclization of thioureas *IIa*, *IIc – IIf* via the nitrogen atom affording 1-(4-substituted phenyl)-4-oxo-2-thioxo-1,3-diazaspiro[5.5]undecanes *IVa*, *IVc – IVf* (Scheme 1) was performed by stirring of an ethanolic solution in the presence of NaOH for several minutes. The recently described[3] cyclization procedure for some α,β-unsaturated acylthioureas in LiH/DMF appeared to be not effective in our case because cyclization is either slow or does not proceed at all.

TABLE I
IR and ^{13}C NMR spectral data of 1,3-thiazines *IIIa – IIIe* and *VI*

Compound	IR, cm^{-1}				^{13}C NMR, ppm	
	ν, C=O		ν, C=N		C=O	C=N
	$CHCl_3$	KBr	$CHCl_3$	KBr		
IIIa	1 698	1 688	1 597	1 598	169.81	150.65
	1 710	1 710	1 620	1 613		
IIIb	1 701	1 710	1 585	1 589	169.72	153.05
			1 617	1 612		
IIIc	1 688	1 672	1 578	1 578	169.69	151.55
	1 698	1 680	1 605	1 612		
IIId	1 690	1 702	1 602	1 608	169.77	150.67
	1 700	1 712	1 618	1 623		
IIIe	1 688	1 700	1 570	1 579	169.80	151.06
	1 698	1 710	1 606	1 605		
VI	1 650	1 665	1 480	1 502	178.34	168.87
	1 662	1 675		1 532		

Based on the literature information[10,11], we supposed that 1,3-diazaspiro[5.5]undecanes *IV* could be obtained also by Dimroth rearrangement from 1,3-thiazaspiro[5.5]undecanes *III*. It appeared, however, that the investigated 1,3-thiazines do not undergo this rearrangement by heating in different solvents, in DMF with presence of LiH, or even after several hour heating in boiling ethanolic NaOH. No other reaction (e.g. hydrolysis) was observed evidencing thus the high stability of 1,3-thiazines *III*.

The presence of a C=S group in the vicinity of a benzene ring in 1,3-diazaspiro[5.5]undecanes *IV* creates the possibility to use these compounds in the synthesis of heterocycles possessing the thiazolo[3,2-*a*]pyrimidine moiety which can be of interest from the point of view of biological activity[12]. According to Garin et al.[13] the intramolecular cyclization can be advantageously accomplished by *N*-halogenosuccinimides in sulfuric acid. In the case of the 1,3-diazaspiro[5.5]undecanes we have found that the most suitable reagent for this cyclization is *N*-bromosuccinimide in concentrated sulfuric acid. Besides the cyclization also the substitution in the position 5 of the pyrimidine ring takes place and 4-bromo-3-oxopyrimido[2,3-*b*]benzothiazol-5-spirocyclohexane *V* is formed as the final product (Scheme 2).



V

SCHEME 2

EXPERIMENTAL

Infrared absorption spectra (wavenumbers in cm^{-1}) were measured on a Specord IR-75 spectrometer (Zeiss, Jena); ^1H NMR spectra were taken on a Tesla BS 487A (80 MHz) and Bruker AMX 300 (300 MHz), ^{13}C NMR spectra on a Tesla 567 (25.15 MHz) and Bruker AMX 300 (75 MHz) instruments; internal standard TMS, δ-values in ppm. Mass spectra were obtained with a MS 902S (AEI Manchester) spectrometer at 70 eV (ionization chamber temperature 190 °C). The reaction course was followed by thin-layer chromatography on Silufol plates (Kavalier).

Cyclohexylideneacetyl Isothiocyanate (*I*)

Lead thiocyanate (1.74 g, 5.5 mmol) was added to a solution of cyclohexylideneacetyl chloride[14] (1.59 g, 10 mmol) in anhydrous benzene (50 ml). After refluxing for 2.5 h, the precipitated $PbCl_2$ was filtered off and the benzene driven off in vacuo, leaving a red-brown oil (85%). IR spectrum ($CHCl_3$): 1 606 (C=C); 1 681 (C=O); 1 953 (N=C=S). ^1H NMR spectrum ($CHCl_3$): 1.62 m, 6 H; 2.15 m, 2 H; 2.80 m, 2 H; 5.95 s, 1 H (C=C–H).

General Procedure for Preparation of *N*-(4-Substituted Phenyl)-*N'*-cyclohexylidene-acetylthioureas *IIa – IIf*

A solution of 4-substituted aniline (10 mmol) in anhydrous benzene or acetone (10 ml) was added dropwise under vigorous stirring to a solution of cyclohexylideneacetyl isothiocyanate *I* (1.81 g, 10 mmol) in anhydrous benzene or acetone (10 ml). After stirring for 30 min the precipitate was filtered off, dried and recrystallized.

N-Phenyl-N'-cyclohexylideneacetylthiourea (IIa), yield 67%, m.p. 118 – 120 °C (ethanol). IR spectrum ($CHCl_3$): 1 515 (NH–CS); 1 631 (C=C); 1 673 (C=O); 3 410 (NH). ^{1}H NMR spectrum ($CDCl_3$): 1.67 m, 6 H; 2.20 m, 2 H; 2.90 m, 2 H; 5.62 s, 1 H (C=C–H); 8.90 s, 1 H (NH). ^{13}C NMR spectrum ($CDCl_3$): 179.10, 168.16, 166.78, 137.77, 130.23, 128.08, 125.43, 122.73, 114.97, 112.69, 38.52, 30.27, 29.23, 27.33, 25.76. Mass spectrum, *m/z* (%): 274 (M^+, 73); 153 (40); 123 (100). For $C_{15}H_{18}N_2OS$ (274.4) calculated: 65.66% C, 6.61% H, 10.21% N; found: 65.38% C, 6.51% H, 10.22% N.

N-(4-Nitrophenyl)-N'-cyclohexylideneacetylthiourea (IIb), yield 71%, m.p. 183 °C (ethanol). IR spectrum ($CHCl_3$): 1 524 (NH–CS); 1 633 (C=C); 1 670 (C=O); 3 415 (NH). For $C_{15}H_{17}N_3O_3S$ (319.4) calculated: 56.41% C, 5.37% H, 13.16% N; found: 56.39% C, 5.31% H, 13.15% N.

N-(4-Bromophenyl)-N'-cyclohexylideneacetylthiourea (IIc), yield 62%, m.p. 122 – 123 °C (ethanol). IR spectrum ($CHCl_3$): 1 520 (NH–CS); 1 630 (C=C); 1 675 (C=O); 3 410 (NH). ^{1}H NMR spectrum ($CDCl_3$): 1.62 m, 6 H; 2.15 m, 2 H; 2.85 m, 2 H; 5.60 s, 1 H (=CH–); 7.52 m, 4 H (C_6H_4); 8.97 bs, 1 H (NH). ^{13}C NMR spectrum ($CDCl_3$): 179.02, 168.68, 166.59, 136.985, 131.91, 125.53, 119.71, 113.70, 38.56, 28.78, 27.96, 26.09. For $C_{15}H_{17}BrN_2OS$ (353.3) calculated: 51.00% C, 4.85% H, 7.93% N; found: 50.98% C, 4.82% H, 7.90% N.

N-(4-Methylphenyl)-N'-cyclohexylideneacetylthiourea (IId), yield 71%, m.p. 169 °C (ethanol). IR spectrum ($CHCl_3$): 1 520 (NH–CS); 1 642 (C=C); 1 676 (C=O); 3 420 (NH). ^{1}H NMR spectrum ($CDCl_3$): 5.72 s, 1 H (–C=CH–); 7.27 m, 4 H (C_6H_5); 9.05 bs, 1 H (NH). ^{13}C NMR spectrum ($CDCl_3$): 179.06, 167.82, 166.63, 136.50, 135.31, 129.38, 124.15, 113.92, 38.45, 30.27, 28.74, 27.92, 26.09, 21.03. For $C_{18}H_{20}N_2OS$ (312.4) calculated: 69.20% C, 6.45% H, 8.97% N; found: 69.15% C, 6.41% H, 8.95% N.

N-(4-Methoxyphenyl)-N'-cyclohexylideneacetylthiourea (IIe), yield 77%, m.p. 103 °C (ethanol). IR spectrum ($CHCl_3$): 1 505 (NH–CS); 1 625 (C=C); 1 670 (C=O); 3 410 (NH). ^{1}H NMR spectrum ($CDCl_3$): 3.80 s, 3 H (OCH_3); 5.62 s, 1 H (–C=CH–); 7.22 m, 4 H (C_6H_4); 9.02 s, 1 H (NH). ^{13}C NMR spectrum ($CDCl_3$): 179.28, 167.82, 166.67, 158.12, 130.83, 125.79, 114.03, 113.88, 55.47, 38.45, 30.27, 28.74, 27.92, 26.09. For $C_{18}H_{20}N_2O_2S$ (328.4) calculated: 65.83% C, 6.14% H, 8.53% N; found: 65.80% C, 6.10% H, 8.51% N.

N-(4-Acetylphenyl)-N'-cyclohexylideneacetylthiourea (IIf), yield 75%, m.p. 174 –176 °C (ethanol). IR spectrum ($CHCl_3$): 1 527 (NH–CS); 1 582 (C=C); 1 638 (CO–NH); 1 678 (CO–CH_3). ^{1}H NMR spectrum (DMSO): 6.42 s, 1 H (=CH–); 8.27 s, 4 H (C_6H_4); 11.60 s, 1 H (NH). ^{13}C NMR spectrum (DMSO): 196.43, 178.89, 166.78, 141.82, 133.83, 128.61, 122.82, 114.09, 29.28, 28.16, 27.30, 26.33, 25.32. For $C_{19}H_{20}N_2O_2S$ (340.4) calculated: 67.03% C, 5.92% H, 8.23% N; found: 67.00% C, 5.88% H, 8.20% N.

General Procedure for Preparation of 2-(4-Substituted Phenylimino)-4-oxo-1,3-thiazaspiro[5.5]undecanes *IIIa – IIIe*

To a solution of thiourea *II* (8 mmol) in a minimum amount of chloroform (10 – 20 ml), boron trifluoride etherate (2.27 g, 16 mmol) is added. After stirring at room temperature for 5 days, the mixture is diluted with chloroform (80 ml) and washed with 4% water solution of sodium bicarbonate (50 ml). The organic layer is separated and the water layer is extracted with chloroform (40 ml). The

combined chloroform extracts are dried with magnesium sulfate and the solid residue remaining after removal of solvents is crystallized.

2-Phenylimino-4-oxo-1,3-thiazaspiro[5.5]undecane (IIIa), yield 83%, m.p. 155 °C (ethanol). IR spectrum ($CHCl_3$): 3 356 (NH). ^{1}H NMR spectrum ($CDCl_3$): 1.50 m, 10 H (C_6H_{10}); 2.82 s, 2 H (CH_2–CO); 7.12 m, 5 H (C_6H_5); 8.32 s, 1 H (NH). ^{13}C NMR spectrum ($CDCl_3$): 169.84, 150.47, 147,03, 128.93, 124.34, 121.61, 48.38, 46.92, 37.59, 25.20, 22.02. For $C_{15}H_{18}N_2OS$ (274.3) calculated: 65.66% C, 6.61% H, 10.21% N; found: 65.52% C, 6.53% H, 10.17% N.

2-(4-Nitrophenylimino)-4-oxo-1,3-thiazaspiro[5.5]undecane (IIIb), yield 59%, m.p. 199 – 202 °C (ethanol). ^{1}H NMR spectrum ($CDCl_3$): 1.53 m, 10 H (C_6H_{10}); 2.83 s, 2 H CH_2–CO); 7.60 m, 4 H (C_6H_4). ^{13}C NMR spectrum ($CDCl_3$): 169.72, 153.05, 152.12, 144.38, 124.92, 122.24, 49.34, 46.74, 37.41, 24.93, 21.88. For $C_{15}H_{17}N_3O_3S$ (319.4) calculated: 56.41% C, 5.37% H, 13.16% N; found: 56.37% C, 5.32% H, 13.12% N.

2-(4-Bromophenylimino)-4-oxo-1,3-thiazaspiro[5.5]undecane(IIIc), yield 77%, m.p. 210 °C (ethanol). ^{1}H NMR spectrum ($CDCl_3$): 1.60 m, 10 H (C_6H_{10}); 2.80 s, 2 H (CH_2–CO); 7.12 m, 4 H (C_6H_4). ^{13}C NMR spectrum ($CDCl_3$): 169.80, 151.51, 145.99, 132.06, 123.55, 117.62, 48.75, 46.92, 37.59, 25.16, 22.02. For $C_{15}H_{17}BrN_2OS$ (353.3) calculated: 51.00% C, 4.85% H, 7.93% N; found: 50.95% C, 4.80% H, 7.91% N.

2-(4-Methylphenylimino)-4-oxo-1,3-thiazaspiro[5.5]undecane (IIId), yield 76%, m.p. 155 – 156 °C (ethanol). ^{1}H NMR spectrum ($CDCl_3$): 1.52 m, 10 H (C_6H_{10}); 2.31 s, 3 H (CH_3); 2.80 s, 2 H (CH_2–CO); 6.90 m, 4 H (C_6H_4). ^{13}C NMR spectrum ($CDCl_3$): 169.77, 150.67, 144.11, 133.91, 129.50, 121.43, 48.30, 46.77, 37.44, 25.07, 21.89, 20.94. For $C_{18}H_{20}N_2OS$ (312.4) calculated: 69.20% C, 6.45% H, 8.97% N; found: 69.17% C, 6.42% H, 8.93% N.

2-(4-Methoxyphenylimino)-4-oxo-1,3-thiazaspiro[5.5]undecane(IIIe), yield 65%, m.p. 132 – 133 °C (ethanol). ^{1}H NMR spectrum ($CDCl_3$): 1.52 m, 10 H (C_6H_{10}); 2.80 s, 2 H (CH_2–CO); 3.77 s, 3 H (OCH_3); 6.86 m, 4 H (C_6H_4); 8.30 s, 1 H (NH). ^{13}C NMR spectrum ($CDCl_3$): 169.80, 156.56, 151.06, 139.75, 122.74, 114.08, 55.30, 48.29, 46.81, 37.45, 25.07, 21.89. For $C_{18}H_{20}N_2O_2S$ (328.4) calculated: 65.83% C, 6.14% H, 8.53% N; found: 65.78% C, 6.11% H, 7.48% N.

General Procedure for Preparation of 1-(4-Substituted Phenyl)-4-oxo-2-thioxo-1,3-diazaspiro[5.5]undecanes *IVa*, *IVc – IVf*

To a solution of the thiourea *II* (10 mmol) in ethanol (80 ml) was added dropwise 2 M NaOH (10 ml). After 15 min water was added and the mixture was neutralized with 2 M HCl. The precipitate was filtered off, dried and crystallized.

1-Phenyl-4-oxo-2-thioxo-1,3-diazaspiro[5.5]undecane(IVa), yield 87%, m.p. 178 – 180 °C (ethanol). IR spectrum ($CHCl_3$): 1 604 (C=N); 1 708 (C=O); 3 380 (NH). ^{1}H NMR spectrum ($CDCl_3$): 1.60 m, 10 H (C_6H_{10}); 2.98 s, 2 H (CH_2–CO); 7.27 m, 5 H (C_6H_5); 9.25 bs, 1 H (NH). ^{13}C NMR spectrum (DMSO): 179.22, 165.90, 140.51, 129.61, 128.61, 127.67, 61.68, 34.13, 24.31, 21.59. For $C_{15}H_{18}N_2OS$ (274.4) calculated: 65.66% C, 6.61% H, 10.21% N; found: 65.59% C, 6.56% H, 10.16% N.

1-(4-Bromophenyl)-4-oxo-2-thioxo-1,3-diazaspiro[5.5]undecane (IVc), yield 78%, m.p. 210 °C (ethanol). IR spectrum ($CHCl_3$): 1 705 (C=O); 3 380 (NH). ^{1}H NMR spectrum ($CDCl_3$): 1.90 m, 10 H (C_6H_{10}); 3.26 s, 2 H (CH_2); 7.65 m, 4 H (C_6H_4); 11.45 bs, 1 H (NH). ^{13}C NMR spectrum (DMSO): 179.33, 165.90, 139.88, 131.97, 131.63, 123.04, 115.58, 61.83, 34.13, 24.35, 21.55. For $C_{15}H_{17}BrN_2OS$ (353.3) calculated: 51.00% C, 4.85% H, 7.93% N; found: 50.97% C, 4.81% H, 7.89% N.

1-(4-Methylphenyl)-4-oxo-2-thioxo-1,3-diazaspiro[5.5]undecane(IVd), yield 80%, m.p. 204 – 206 °C (ethanol). IR spectrum (KBr): 1 718 (C=O); 3 380 (NH). ^{1}H NMR spectrum ($CDCl_3$): 1.61 m, 10 H

(C_6H_{10}); 2.48 s, 3 H (CH_3); 3.02 s, 2 H (CH_2); 7.27 m, 4 H (C_6H_4); 9.20 bs, 1 H (NH). ^{13}C NMR spectrum ($CDCl_3$): 179.54, 165.28, 138.67, 137.81, 130.05, 129.15, 62.97, 38.71, 34.98, 22.47, 21.23. For $C_{18}H_{20}N_2OS$ (312.4) calculated: 69.20% C, 6.45% H, 8.97% N; found: 69.15% C, 6.39% H, 8.92% N.

1-(4-Methoxyphenyl)-4-oxo-2-thioxo-1,3-diazaspiro[5.5]undecane (IVe), yield 81%, m.p. 235 – 237 °C (ethanol). IR spectrum ($CHCl_3$): 1 710 (C=O); 3 380 (NH). ^{1}H NMR spectrum ($CDCl_3$): 1.37 m, 10 H (C_6H_{10}); 2.32 s, 2 H (CH_2); 3.85 s, 3 H (CH_3); 7.07 m, 4 H (C_6H_4); 9.07 bs, 1 H (NH). ^{13}C NMR spectrum ($CDCl_3$): 179.84, 165.25, 159.50, 133.18, 130.42, 114.56, 63.08, 55.43, 38.75, 34.98, 24.75, 22.51. For $C_{18}H_{20}N_2O_2S$ (328.4) calculated: 65.83% C, 6.14% H, 8.53% N; found: 65.80% C, 6.09% H, 8.50% N.

1-(4-Acetylphenyl)-4-oxo-2-thioxo-1,3-diazaspiro[5.5]undecane (IVf), yield 62%, m.p. 230 – 233 °C (ethanol). IR spectrum (KBr): 1 687 (NH–C=O); 1 738 (CH_3C=O). ^{1}H NMR spectrum (DMSO): 1.62 m, 10 H (C_6H_{10}); 2.62 s, 3 H (CH_3); 2.90 s, 2 H (CH_2); 7.97 m, 4 H (C_6H_4). ^{13}C NMR spectrum (DMSO): 197.03, 179.11, 165.90, 144.70, 135.92, 130.17, 129.39, 128.64, 120.80, 62.01, 34.17, 26.66, 24.39, 21.55. For $C_{19}H_{20}N_2O_2S$ (340.4) calculated: 67.03% C, 5.92% H, 8.23% N; found: 66.99% C, 5.87% H, 8.22% N.

4-Bromo-3-oxopyrimido[2,3-*b*]benzothiazol-5-spirocyclohexane (*V*)

A solution of *IVa* (0.549 g, 2 mmol) and *N*-bromosuccinimide (0.356 g, 2 mmol) in concentrated sulfuric acid (6 ml) was heated with stirring for 5 h. The solution was then cooled, poured into water (50 ml) in an ice-water bath and made alkaline (pH 9) by dropwise addition of 20% aqueous sodium hydroxide. The resulting precipitate was washed with 5% aqueous sodium hydroxide and then with water, dried and recrystallized from ethanol. Yield 0.486 g (69%) of compound *V*, m.p. 221 °C (ethanol). IR spectrum ($CHCl_3$): 1 672 (C=O). ^{1}H NMR ($CDCl_3$): 4.91 s, 1 H (CH–Br). Mass spectrum, *m*/*z* (%): 352 (M + 1, 5), 350 (5), 273 (44), 272 (44), 271 (20), 176 (100). For $C_{15}H_{16}BrN_2OS$ (352.3) calculated: 51.14% C, 4.58% H, 7.95% N; found: 51.08% C, 4.54% H, 7.89% N.

2-(*N*-Methylphenylamino)-4-oxo-1,3-thiazaspiro[5.5]undec-2-ene (*VI*)

A solution of *N*-methylaniline (0.65 ml, 6 mmol) in acetone (6 ml) was added at room temperature to isothiocyanate *I* (1.09 g, 6 mmol) in the same solvent (10 ml) and the mixture was stirred for 25 min. The oil obtained after evaporating of solvent was chromatographed on silica gel (eluent benzene–acetone 5 : 2) before crystallization from 1-propanol. Yield 1.28 g (74%) of compound *VI*, m.p. 154 – 156 °C (ethanol). ^{1}H NMR spectrum ($CDCl_3$): 1.60 m, 10 H (C_6H_{10}); 2.54 s, 2 H (CH_2); 3.45 s, 3 H (N–CH_3); 7.28 m, 5 H (C_6H_5). ^{13}C NMR spectrum ($CDCl_3$): 178.34, 169.87, 142.35, 129.69, 128.85, 127.85, 50.57, 44.78, 40.64, 37.38, 25.09, 21.73. For $C_{16}H_{21}N_2OS$ (289.4) calculated: 66.40% C, 7.31% H, 9.68% N; found: 66.35% C, 7.28% H, 9.61% N.

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