

New Schiff bases from 2,5-bis-(butylsulfanyl)-2,3-dihydro-4H-pyran-2-carbaldehyde

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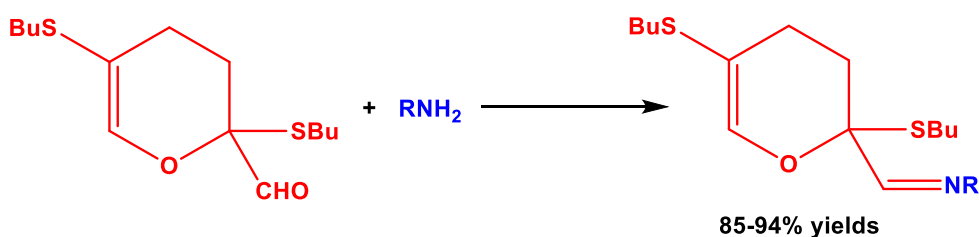
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
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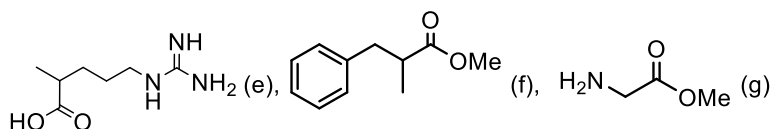
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Abstract

Novel densely functionalized Schiff bases have been synthesized by the reaction of 2,5-bis-(butylsulfanyl)-2,3-dihydro-4H-pyran-2-carbaldehyde with primary amines and amino acids. Structure of the obtained compounds is confirmed using the data of elemental analysis, IR and NMR techniques, and chromatomass-spectrometry.



R = Bu (a), CH₂Ph (b), Ph (c),  (d)



Keywords: Schiff bases, azomethines, 2,5-bis-(butylsulfanyl)-2,3-dihydro-4H-pyran-2-carbaldehyde, aldehydes, primary amines, amino acids

Introduction

Schiff bases, also known as imines or azomethines, represent an important class of organic compounds, which originate from condensation of primary amines and active carbonyls.^{1,2} Schiff bases are among the most widely employed chemical substances that find application as pigments and dyes, temperature sensors, corrosion inhibitors, catalysts for many reactions, intermediates in organic synthesis and ligands in coordination chemistry.³⁻⁹ Schiff bases also exhibit a wide range of biological activity, including antibacterial, hypotensive, antifungal, antipyretic, anti-tumor, anti-inflammatory and anti-HIV.^{10,11}

Derivatives of Schiff bases with amino acids,^{12,13} building blocks for the synthesis of proteins and polypeptides^{14,15} and various heterocyclic compounds^{16,17} are of particular interest. The complexes of amino acid with Schiff bases have gained distinction due to their physiological and pharmacological activity.¹⁸⁻²⁰ These are very attractive compounds that mimic metal enzymes found in living organisms.^{21,22}

Dihydropyrans are important intermediates for the synthesis of biologically active natural products such as kendomycin, ambruticin and leucascandrolide A (Figure 1).²³⁻³⁵ The 4-methyl substituted dihydropyran unit is present in the macrolide natural products laulimalide and okadaic acid.^{36,37} Substituted dihydropyrans are also used as a flavoring or aroma material for food and other products.³⁸

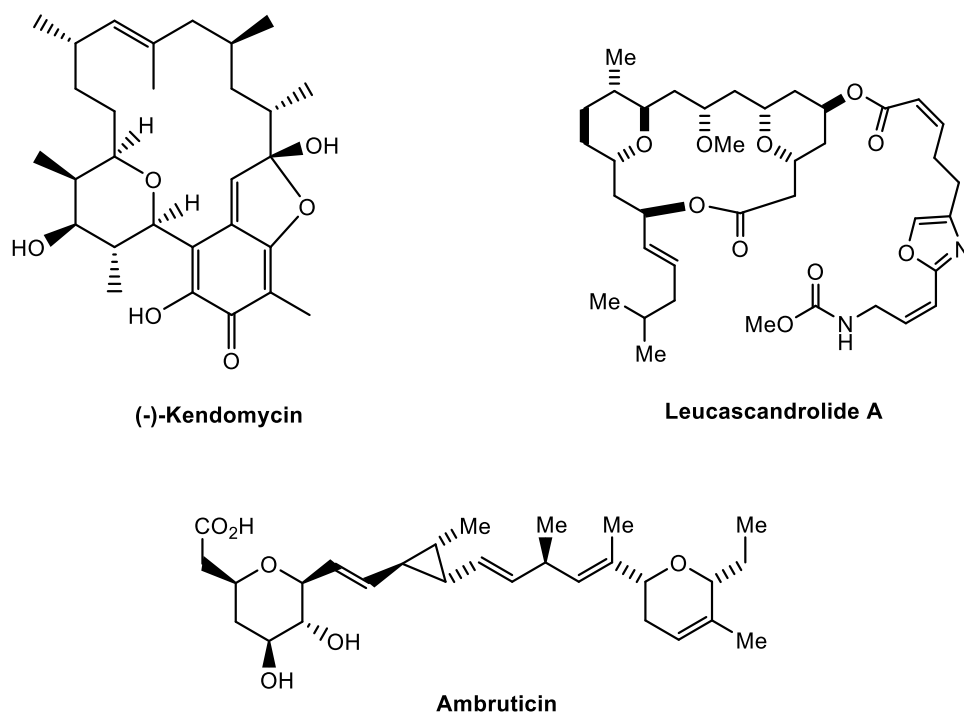


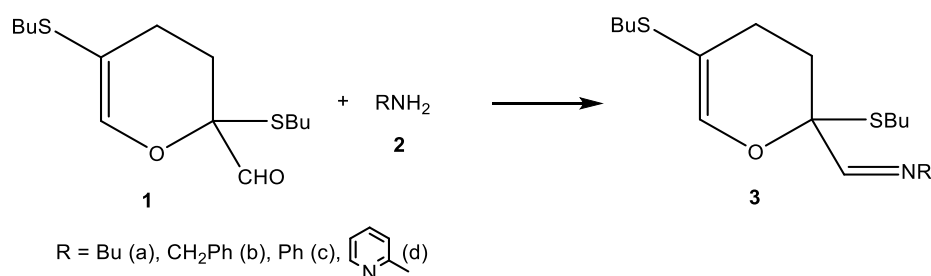
Figure 1. Natural products bearing dihydropyran moiety.

Imine derivatives of L-arginine with heteroaromatic aldehydes are known to exhibit antioxidant activity.³⁹ 2,5-Bis-(butylsulfanyl)-2,3-dihydro-4*H*-pyran-2-carbaldehyde (**1**) is claimed to be a strong and safe antiseptic.⁴⁰ In order to obtain of hitherto unknown Schiff bases from aldehyde **1**, we studied its interaction with primary amines, as well as amino acids and their esters.

Results and Discussion

It is found (Scheme 1) that pyran **1** reacts with an equimolar amount of butylamine **2a** in THF at 24 °C for 1 h (Table 1, entry 1) to afford azomethine **3a** in almost quantitative yield (^1H NMR), the conversion of aldehyde **1** being complete. Condensation of aldehydes with primary amines is an easily reversible reaction. The reaction mixture contains enough moisture to allow hydrolysis of compound **3**. To prevent this process, drying agents (molecular sieves 4A, MgSO_4) are employed. Benzylamine (**2b**) reacts similarly under these conditions (Table 1, entry 2). Filtration of the reaction mixture and evaporation of the solvent in vacuo leads to imines that do not require additional purification.

Schiff bases **3a** and **3b** were obtained as single *E* isomer. The NOESY spectra show cross peaks of the CHN group proton with the CH_2 protons in the substituent at nitrogen. In the ^1H NMR spectra, there is one signal belonging to the $\text{CH} = \text{N}$ proton.



Scheme 1. Reaction of 2,5-bis-(butylsulfanyl)-2,3-dihydro-4*H*-pyran-2-carbaldehyde with primary amines.

Aniline **2c** and 2-aminopyridine **2d** do not interact with pyran **1** under comparable conditions (24 °C, 2 h) owing to significant decrease of their nitrogen atoms basicity (Table 1, entry 3, 5). A longer reaction time and other solvent also does not give the desired result (Table 1, entry 6, 7). Even upon refluxing in MeOH, the starting substrate **2d** does not react (Table 1, entry 8). In the case of aniline **2c**, changing the solvent made it possible to obtain the desired product in 85% yield (Table 1, entry 4).

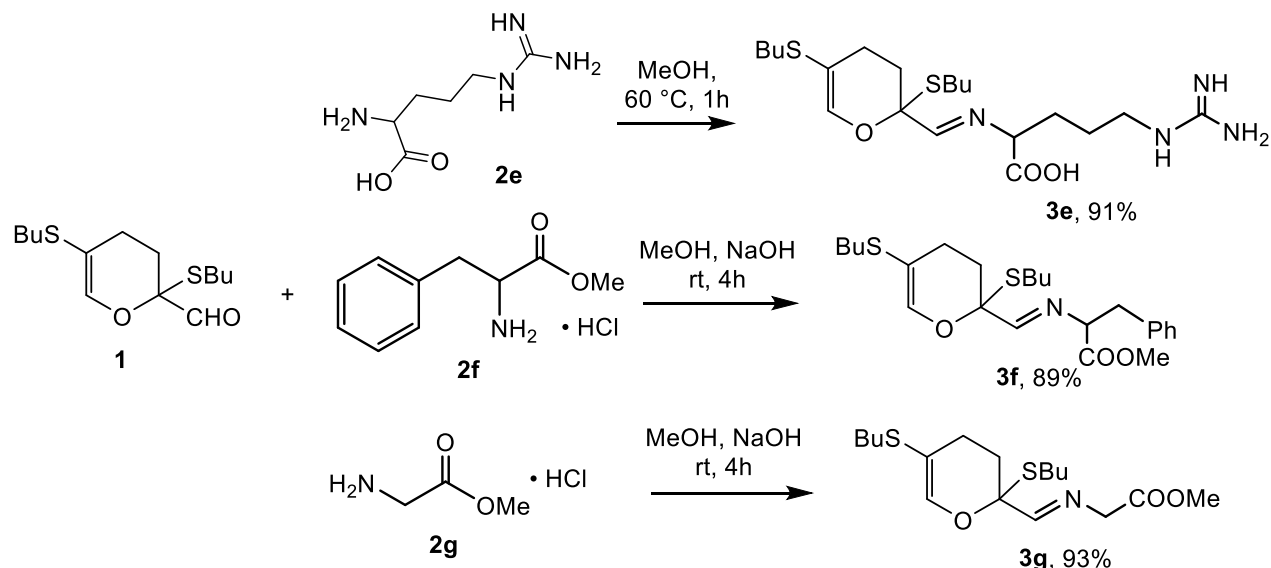
Table 1. Condensation of 2,5-dibutylthio-2,3-dihydro-2-formyl-4*H*-pyran (**1**) with primary amines **2a-d**: the reaction conditions^a

Entry	Amine	Solvent	Temp.(°C)	<i>t</i> (h)	NMR ^1H yield 3 (%)
1	2a	THF	24	1	98 (94 ^b)
2	2b	THF	24	1	97 (89 ^b)
3	2c	THF	24	2	-
4	2c	MeOH	reflux	6	97 (85 ^b)
5	2d	THF	24	2	-
6	2d	THF	24	48	-
7	2d	MeOH	24	48	-
8	2d	MeOH	reflux	48	-

^a Pyran **1** (1 mmol), amine **2** (1 mmol), MeOH/THF (1 ml). ^b Isolated yield.

The imine derivatives of L- arginine have been obtained according to the method published previously.³⁹

The reaction with arginine **2e** occurs upon boiling in methanol for 1 h to deliver the iminium derivative **3e** in 91% yield (Scheme 2).



Scheme 2. The reaction of 2,5-bis-(butylsulfanyl)-2,3-dihydro-4H-pyran-2-carbaldehyde (**1**) with amino acids and their esters **2e-g**.

Owing to limited solubility in organic solvents and reduced basicity of glycine and phenylalanine, such conditions turn out to be inappropriate. 2,5-Bis-(butylsulfanyl)-2,3-dihydro-4H-pyran-2-carbaldehyde (**1**) reacted with α -amino acid (DL phenylalanine and glycine) methyl esters **2f**, **2g** (generated *in situ* from the hydrochlorides by reaction of NaOH) in MeOH at room temperature to give previously unknown Schiff bases **3f**, **3g** in 89-93% yield (Scheme 2).

The ¹H NMR spectrum of Schiff bases **3e,f** shows a mixture of diastereomers (relative to chiral centers in the parent aldehyde and amino acid (L-arginine and phenylalanine)), existing in the form of an *E*-configuration (according to NOESY), as indicated by the value of the spin-spin coupling constant (¹J_{CH} = 166-167 Hz,) of the carbon fragment HC=N.⁴¹

Conclusions

In conclusion, we have reported the facile and valuable synthesis of functionalized imine derivatives of original 2,5-bis-(butylsulfanyl)-2,3-dihydro-4H-pyran-2-carbaldehyde with primary amines and amino acids. Using this protocol, we were able to assemble a range of functionalized Schiff bases that represent a promising and interesting application in construction of metallocomplexes.

Experimental Section

General. The ^1H , ^{13}C and ^{15}N NMR spectra were recorded in CDCl_3 solutions at room temperature on Bruker DPX-400 and AV-400 spectrometers (400.13, 100.61 and 40.56 MHz, respectively). ^1H , ^{13}C and ^{15}N Chemical shifts (δ in ppm) were measured with accuracy of 0.01, 0.02 and 0.1 ppm, respectively, and referred to TMS (^1H , ^{13}C) and nitromethane (^{15}N). Chromato-mass spectrometry analysis was performed on a Shimadzu GCMS-QP5050A mass spectrometer (EI ionization, 70 eV). The IR spectra of the compounds were recorded on a Varian 3100 FT-IR spectrometer with the sample in thin film. Elemental analysis was performed on a Thermo Finnigan Flash series 1112 Elemental analyzer.

General synthesis of compounds 3a-c, e. Compounds **3a-c, e**: To the solution of **1** (1 mmol, 0.288 g) of compound (**1**) in MeOH or THF was added 1 mmol of amine (**2a-c**) or amino acid (**2e**) and the mixture was stirred for 1-6 h at room temperature or reflux. The reaction mixture was dried with MgSO_4 , the precipitate was filtered off and the solvent was removed in vacuo. The desired imine was obtained as an oil or powder.

N-{(1E)-[2,5-bis-(butylthio)-3,4-dihydro-2H-pyran-2-yl]methylen}butylamine (3a). Dark brown oil, yield 94%. IR, ν , (cm^{-1}): 2953, 2929, 2865, 1627, 1458, 1145, 1046, 768. ^1H NMR (CDCl_3) δ : 0.89-0.94 (m, 9H, 3CH_3 in 2SBu and NBu), 1.32-1.40 (m, 6H, CH_2CH_3 in 2SBu and NBu), 1.49-1.52 (m, 4H, $2\text{SCH}_2\text{CH}_2$ in 2SBu), 1.57-1.61 (m, 2H, NCH_2CH_2), 2.09-2.11 (m, 2H, CH_2 at C-4), 2.19 and 2.44 (two m, 2H, CH_2 at C-3), 2.43-2.56 (m, 4H, 2SCH_2 in 2SBu), 3.42-3.53 (m, 2H, NCH_2), 6.67 (s, 1H, =CH), 7.51 (s, 1H, CH=N). ^{13}C NMR (CDCl_3): 13.72 (CH_3 in NBu), 13.82 (CH_3 in SBu at C*), 13.93 (CH_3 in SBu at C=), 20.47 (CH_2CH_3 in NBu), 21.74 (CH_2CH_3 in SBu at C*), 22.21 (CH_2CH_3 in SBu at C=), 23.90 (C-3), 27.52 (SCH_2 at C*), 29.86 (C-4), 31.60 (SCH_2CH_2 at C*), 31.92 (SCH_2CH_2 at C=), 32.46 (SCH_2 at C=), 32.92 (NCH_2CH_2), 60.68 (NCH_2), 84.61 (C*), 107.27 (C-5), 144.10 (=CHO), 160.79 (CH=N). ^{15}N NMR (CDCl_3): - 56.4. MS-HRMS m/z , (J_{OTH} , %): 343 (1) $[\text{M}]^+$, 254 (15) $[\text{M-SBu}]^+$, 213 (3), 199 (3), 185 (4), 171 (3), 164 (4), 149 (6), 129 (9), 115 (7), 98 (8), 85 (67), 83 (100), 69 (65), 43 (63), 41 (77). Found (%): C, 63.03; H, 9.73; N, 4.12; S, 18.52. Calculated $\text{C}_{18}\text{H}_{33}\text{NOS}_2$ (%): C, 62.92; H, 9.68; N, 4.08; S, 18.66.

N-{(1E)-[2,5-bis-(butylthio)-3,4-dihydro-2H-pyran-2-yl]methylen}benzylamine (3b). Dark brown oil, yield 89%. IR, ν , (cm^{-1}): 2956, 2927, 2866, 1665, 1627, 1455, 1145, 1095, 1046, 738, 698. ^1H NMR (CDCl_3) δ : 0.87 (t, 3H, CH_3 (SBu at C*), $J = 7.5$ Hz), 0.92 (t, 3H, CH_3 (SBu at C=), $J = 7.3$ Hz), 1.31 (q, 2H, CH_2CH_3 (SBu at C*), $J = 7.5$ Hz), 1.40 (q, 2H, CH_2CH_3 (SBu at C=), $J = 7.3$ Hz), 1.44-1.49 (m, 2H, SCH_2CH_2 (SBu at C*)), 1.50-1.56 (m, 2H, SCH_2CH_2 (SBu at C=)), 2.14-2.17 (m, 2H, CH_2 at C-4), 2.21 and 2.45 (two m, 2H, CH_2 at C-3), 2.49-2.55 (m, 4H, 2SCH_2), 4.66 and 4.73 (two d, 2H, CH_2Ph , $J = 13.5$ Hz), 6.66 (s, 1H, =CH), 7.23-7.34 (m, 5H, Ph), 7.65 (s, 1H, CH=N). ^{13}C NMR (CDCl_3): 13.81 (CH_3 in SBu at C*), 13.95 (CH_3 in SBu at C=), 21.81 (CH_2CH_3 in SBu at C*), 22.24 (CH_2CH_3 in SBu at C=), 23.96 (C-3), 27.60 (SCH_2 in SBu at C*), 29.70 (C-4), 31.65 (SCH_2CH_2 in SBu at C*), 31.99 (SCH_2CH_2 in SBu at C=), 32.47 (SCH_2 in SBu at C=), 64.32 (NCH_2), 84.74 (C*), 107.59 (C-5), 127.33 (C_p), 128.21 (C_m), 128.67 (C_o), 138.69 (C_{ipso}), 143.95 (=CHO), 162.13 (CH=N). ^{15}N NMR (CDCl_3): - 59.8. MS-HRMS m/z , (J_{OTH} , %): 377 (1) $[\text{M}]^+$, 348 (5), 288 (58) $[\text{M-SBu}]^+$, 231 (2), 198 (14), 144 (18), 91 (100) $[\text{PhCH}_2]^+$, 65 (7), 55 (4), 41 (13). Found (%): C, 66.93; H, 8.13; N, 3.67; S, 16.91. Calculated $\text{C}_{21}\text{H}_{31}\text{NOS}_2$ (%): C, 66.84; H, 8.22; N, 3.71; S, 16.98.

N-{(1E)-[2,5-bis-(butylthio)-3,4-dihydro-2H-pyran-2-yl]methylen}aniline (3c). Dark brown oil, yield 85%. IR, ν , (cm^{-1}): 2957, 2928, 2868, 1730, 1627, 1490, 1144, 1088, 1044, 758, 694. ^1H NMR (CDCl_3) δ : 0.88-0.93 (m, 6H, 2CH_3 in SBu), 1.36-1.43 (m, 4H, CH_2CH_3), 1.51-1.57 (m, 4H, SCH_2CH_2), 2.23-2.26 (m, 2H, CH_2 at C-4), 2.27 and 2.49 (two m, 2H, CH_2 at C-3), 2.52 (m, 2H, SCH_2 in SBu at C*), 2.63 (m, 2H, SCH_2 in SBu at C=), 6.71 (s, 1H, =CH), 7.09 (d, 2H, $o\text{-H}$, $J = 7.6$ Hz), 7.22 (t, 1H, $p\text{-H}$, $J = 7.4$ Hz), 7.36 (dd, 2H, $m\text{-H}$, $J = 7.6$, $J = 7.4$ Hz), 7.70 (s, 1H, CH=N). ^{13}C NMR (CDCl_3): 13.74 (CH_3 in SBu at C*), 13.83 (CH_3 in SBu at C=), 21.78 (CH_2CH_3 in SBu at C*), 22.23 (CH_2CH_3 in SBu at C=), 23.91 (C-3), 27.64 (SCH_2 at C*), 29.59 (C-4), 31.66 (SCH_2CH_2 at C*), 31.97 (SCH_2CH_2 at C=), 32.48 (SCH_2 at C=).

C=), 84.92 (C*), 107.73 (C-5), 120.94 (C_m); 126.38 (C_p); 129.30 (C_o); 143.91 (C_i), 150.87 (=CHO), 160.7 (CH=N). ¹⁵N NMR (CDCl₃): -61.2. Found (%): C, 66.02; H, 7.85; N, 3.81; S, 17.51. Calculated C₂₀H₂₉NOS₂ (%): C, 66.11; H, 7.99; N, 3.86; S, 17.63.

N²-{[(1E)-[2,5-bis-(butylthio)-3,4-dihydro-2H-pyran-2-yl]methylen}arginine (3e). Gray powder, yield 91%. Compound **3e** is a mixture of diastereomers (*E*-configuration) in 2:1 ratio. IR, ν, (cm⁻¹): 3314, 3264, 2957, 1660, 1627, 1587, 1377, 1145, 1043, 829, 746, 604. ¹H NMR (CD₃OD): **major-3e**: δ 0.95 (m, 6H, 2CH₃ in 2SBu), 1.40-1.47 (m, 4H, 2CH₂CH₃ in 2SBu), 1.51-1.60 (m, 4H, 2SCH₂CH₂ in 2SBu), 1.65 (m, 2H, CH₂CH₂NH), 1.93 (m, 2H, CH*), 2.19 (m, 2H, CH₂ at C-4), 2.24 and 2.48 (two m, 2H, CH₂ at C-3), 2.66 (m, 4H, 2SCH₂ in 2SBu), 3.22 (m, 2H, CH₂NH), 3.79 (m, 1H, CH*COOH), 4.89 (s, 5H, NH, NH₂, OH), 6.65 (s, 1H, =CH), 7.64 (s, 1H, CH=N); **minor-3e**: δ 0.92 (m, 6H, 2CH₃ in 2SBu), 1.36-1.40 (m, 4H, 2CH₂CH₃ in 2SBu), 1.48-1.53 (m, 4H, 2SCH₂CH₂ in 2SBu), 1.61 (m, 2H, CH₂CH₂NH), 1.87 (m, 2H, CH*), 2.11 (m, 2H, CH₂ at C-4), 2.25 and 2.46 (two m, 2H, CH₂ at C-3), 2.61 (m, 4H, 2SCH₂ in 2SBu), 3.20 (m, 2H, CH₂NH), 3.80 (m, 1H, CH*COOH), 4.89 (br. s, 5H, NH, NH₂, OH), 6.64 (s, 1H, =CH), 7.66 (s, 1H, CH=N). ¹³C NMR (CD₃OD): **major-3e**: 14.07 (2CH₃ in 2SBu), 22.55 (2CH₂CH₃ in 2SBu), 24.80 (C-3), 27.06 (2CH₂CH₂CH₃ in 2SBu), 28.39 (C-4), 30.73 (SCH₂ in SBu at C*), 32.20 (SCH₂ in SBu at C=), 32.69 (CH₂CH₂NH), 32.90 (CH₂CH₂CH₂NH), 42.22 (CH₂NH), 76.02 (CH*), 85.81 (C-2), 107.58 (C-5), 144.69 (=CHO), 159.30 (C=NH), 163.06 (CH=N, ¹J_{CH} = 167.0 Hz), 180.50 (COOH); **minor-3e**: 14.11 (CH₃ in SBu), 23.02 (CH₂CH₃ in SBu), 24.80 (C-3), 27.06 (2CH₂CH₂CH₃ in 2SBu), 28.39 (C-4), 30.73 (SCH₂ in SBu at C*), 32.20 (SCH₂ in SBu at C=), 32.69 (CH₂CH₂NH), 33.00 (CH₂CH₂CH₂NH), 42.22 (CH₂NH), 76.02 (CH*), 85.8 (C-2), 107.58 (C-5), 144.68 (=CHO), 159.26 (C=NH), 163.07 (CH=N, ¹J_{CH} = 166.0 Hz), 180.50 (COOH). ¹⁵N NMR (CDCl₃): **major-3e**: -58.3 (CH=N). Found (%): C, 54.17; H, 8.21; N, 12.52, S, 14.32. Calculated C₂₀H₃₆N₄O₃S₂ (%): C, 54.05; H, 8.12; N, 12.61, S, 14.41.

General synthesis of compounds 3f, g. To the solution of **1** (1 mmol, 0.288 g) of compound (**1**) in MeOH was added 1 mmol of amino acid methyl ester hydrochloride (**2f, g**) and NaOH (1 mmol) and the mixture was stirred for 4 h at room temperature. The reaction mixture was dried with MgSO₄, the precipitate was filtered off and the solvent was removed in vacuum. The desired imine was obtained as an oil.

Methyl-N-[(2,5-bis-(butylthio)-3,4-dihydro-2H-pyran-2-yl)methylen]phenyl-alaninate (3f).

Dark brown oil, yield 89%. Compound **3f** is a mixture of diastereomers (*E*-configuration) in ratio 1.3:1. ¹H NMR (DMSO): **major-3f**: δ 0.84 (m, 6H, 2CH₃ in 2SBu), 1.25-1.32 (m, 4H, CH₂CH₃ in 2SBu), 1.37-1.48 (m, 4H, CH₂CH₂CH₃ in 2SBu), 2.01-2.17 (m, 2H, CH₂ at C-4), 2.19-2.22 (m, 2H, CH₂ at C-3), 2.31 (m, 4H, 2SCH₂), 2.93 (m, 2H, CH₂Ph), 3.61 (s, 3H, OMe), 4.25 (two d, 2H, *CH, *J* = 13.5 Hz), 6.60 (s, 1H, =CH), 7.13-7.18 (m, 5H, Ph), 7.41 (s, 1H, CH=N); **minor-3f**: δ 0.80 (t, 6H, 2CH₃ in 2SBu), 1.20-1.24 (m, 4H, CH₂CH₃), 1.32-1.37 (m, 4H, CH₂CH₂CH₃ in 2SBu), 1.84-1.91 (m, 2H, CH₂ at C-4), 2.22-2.25 (m, 2H, CH₂ at C-3), 2.47 (m, 4H, 2SCH₂), 3.19 (m, 2H, CH₂Ph), 3.62 (s, 3H, OMe), 4.31 (two d, 2H, *CH, *J* = 13.5 Hz), 6.55 (s, 1H, =CH), 7.22-7.26 (m, 5H, Ph), 7.53 (s, 1H, CH=N). ¹³C NMR (DMSO): **major-3f**: δ 13.36 (2CH₃ in 2SBu), 21.35 (2CH₂CH₃ in 2SBu), 23.02 (C-3), 26.51 (2CH₂CH₂CH₃ in 2SBu), 28.85 (C-4), 30.91 (SCH₂ in SBu at C*), 31.17 (SCH₂ in SBu at C=), 38.51 (CH₂Ph), 51.85 (OCH₃), 71.87 (*CH), 84.15 (C-2), 106.70 (C-5), 126.35 (C_p), 128.14 (C_o), 129.28 (C_m), 137.15 (C_i), 143.25 (CH=), 164.08 (CH=N, ¹J_{CH} = 165.8 Hz), 171.02 (COOMe); **minor-3f**: δ 13.49 (2CH₃ in 2SBu), 21.42 (2CH₂CH₃ in 2SBu), 23.07 (C-3), 26.61 (2CH₂CH₂CH₃ in 2SBu), 28.86 (C-4), 31.16 (SCH₂ in SBu at C*), 31.23 (SCH₂ in SBu at C=), 38.51 (CH₂Ph), 48.54 (OCH₃), 72.01 (*CH), 84.44 (C-2), 106.75 (C-5), 126.35 (C_p), 128.47 (C_o), 129.40 (C_m), 137.15 (C_i), 143.11 (CH=), 164.64 (CH=N, ¹J_{CH} = 165.8 Hz), 170.93 (COOMe). ¹⁵N NMR (DMSO): **major-3f**: -61.2; **minor-3f**: -60.6. MS-HRMS *m/z*, (*J*_{OTH}, %): 449 (3) [M]⁺, 360 (100) [M-SBu]⁺, 270 (36), 158 (15), 91 (53), 77 (5). Found (%): C, 64.21; H, 7.87; N, 3.01, S, 14.17. Calculated C₂₄H₃₅NO₃S₂ (%): C, 64.14; H, 7.79; N, 3.12, S, 14.25.

Methyl-N-[(2,5-bis-(butylthio)-3,4-dihydro-2H-pyran-2-yl)methylen]glycinate (3g).

Dark brown oil, yield 93%. IR, ν, (cm⁻¹): 2971, 2926, 2831, 1667, 1455, 1131, 1095, 1046, 681. ¹H NMR (DMSO) δ: 0.82-0.87 (m, 6H, CH₃ in SBu), 1.29-1.37 (m, 4H, CH₂CH₃), 1.42-1.47 (m, 4H, SCH₂CH₂), 2.04-2.14 (m, 2H, CH₂ at C-

4), 2.18 and 2.27 (two m, 2H, CH₂ at C-3), 2.57 (m, 4H, 2SCH₂), 3.64 (s, 3H, OMe), 4.26 and 4.32 (d, 2H, CH₂COOMe, $J_{HH} = 16$ Hz), 6.63 (s, 1H, =CH), 7.71 (s, 1H, CH=N). ¹³C NMR (DMSO): 13.35 (CH₃ in SBu at C*), 13.47 (CH₃ in SBu at C=), 20.85 (CH₂CH₃ in SBu at C*), 21.36 (CH₂CH₃ in SBu at C=), 23.14 (C-3), 26.73 (SCH₂ at C*), 28.75 (C-4), 30.86 (SCH₂CH₂ at C*), 31.10 (SCH₂CH₂ at C=), 31.27 (SCH₂ at C=), 51.60 (OMe), 59.48 (NCH₂), 84.43 (C*), 106.83 (C-5), 143.18 (=CHO), 165.3 (CH=N), 169.80 (C=O). ¹⁵N NMR (DMSO): -69.8. MS-HRMS m/z ($J_{OTH.}$, %): 359 (2) [M]⁺, 330 (13), 270 (100) [M-SBu]⁺, 180 (32), 127 (23), 73 (13), 41 (54). Found (%): C, 56.77; H, 8.11; N, 3.99, S, 17.91. Calculated C₁₇H₂₉NO₃S₂ (%): C, 56.82; H, 8.08; N, 3.90, S, 17.83.

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Supplementary Material

Supplementary material is available on the publisher's website along with the published article.

References

1. Tadavi, S. K.; Yadav, A. A.; Bendre, R. S. *J. Mol. Struct.* **2018**, *1152*, 223, <https://doi.org/10.1016/j.molstruc.2017.09.112>.
2. Adamovich, S. N.; Vchislo, N. V.; Oborina, E. N.; Ushakov, I. A.; Rozentsveig, I. B. *Mend. Commun.* **2017**, *27* (5), 443, <https://doi.org/10.1016/j.mencom.2017.09.003>.
3. Alkan, S.; Telli, F. C.; Salman, Y.; Astley, S. T. *Carbohydr. Res.* **2015**, *407*, 97, <https://doi.org/10.1016/j.carres.2015.01.023>.
4. Li, B.; Li, Y.-Q.; Zheng, J. *Arkivoc* **2010** (ix), 163 <https://doi.org/10.3998/ark.5550190.0011.915>.
5. Fang, Z.; Cao, C. *J. Mol. Struct.*, **2013**, *1036*, 447 <https://doi.org/10.1016/j.molstruc.2012.12.014>.
6. Kolcu, F.; Erdener, D.; Kaya, İ. *Inorg. Chim. Acta*, **2020**, *509*, 119676, <https://doi.org/10.1016/j.ica.2020.119676>.
7. Salman, Y.; Barlas, F. B.; Yavuz, M.; Kaya, K.; Timur, S.; Telli, F. Ç. *Inorg. Chim. Acta*, **2018**, *483*, 98, <https://doi.org/10.1016/j.ica.2018.08.010>.
8. Kołodziej, B.; Grech, E.; Schilf, W.; Kamien'ski, B.; Pazio, A.; Woz'niak, K. *J. Mol. Structure*, **2014**, *1063*, 145, <https://doi.org/10.1016/j.molstruc.2014.01.058>.
9. Moosavi-Zare, A. R.; Goudarziafshar, H.; Nooraei, F. *Appl. Organometal. Chem.*, **2020**; *34*, e5252, <https://doi.org/10.1002/aoc.5252>.
10. Unver, Y.; Sancak, K.; Çelik, F.; Birinci, E.; Küçük, M.; Soylu, S.; Arslan, B. N. *Eur. J. Med. Chem.*, **2014**, *84*, 639

- <https://doi.org/10.1016/j.ejmech.2014.01.014>.
11. Süleymanoğlu, N.; Demir, E. E.; Direkel, Ş; Ünver, Y. *J. Mol. Structure*, **2020**, 1218, 128522, <https://doi.org/10.1016/j.molstruc.2020.128522>.
 12. Rozwadowski, Z. *J. Mol. Struct.*, **2005**, 753, 127 <https://doi.org/10.1016/j.molstruc.2005.06.005>.
 13. Matsumoto, Y.; Sawamura, J.; Murata, Y.; Nishikata, T.; Yazaki, R.; Ohshima, T. *J. Am. Chem. Soc.*, **2020**, 142 (18), 8498 <https://doi.org/10.1021/jacs.0c02707>;
 14. Conti, P.; Tamborini, L.; Pinto, A.; Blondel, A.; Minoprio, P.; Mozzarelli, A.; De Micheli, C. *Chem. Rev.*, **2011**, 111, 6919 <https://doi.org/10.1021/cr2000702>.
 15. Fu, X.; Li, Z.; Wei, J.; Sun, J.; Li, Z. *Polym. Chem.*, **2018**, 9, 4617 <https://doi.org/10.1039/C8PY00924D>.
 16. Heravi, M. M.; Vavsari, V. F. in *Advances in Heterocyclic Chemistry*, eds. F.V.E. Scriven and C. A. Ramsden, **2015**, 114, 77 <https://doi.org/10.1016/bs.aihch.2015.02.002>.
 17. Kadem, K. J.; Munahi, M. G. *J. Pharm. Sci. Res.*, **2018**, 10 (6), 1613.
 18. Abdel-Rahman, L. H.; El-Khatib, R. M.; Nassr, L. A. E.; Abu-Dief, A. M. *J. Mol. Struct.*, **2013**, 1040, 9, <https://doi.org/10.1016/j.molstruc.2013.02.023>.
 19. Pervaiz, M.; Ahmad, I.; Yousaf, M.; Kirn, S.; Munawar, A.; Saeed, Z.; Adnan, A.; Gulzar, T.; Kamal, T.; Ahmad, A. *Spectrochim. Acta Part A: Molecular and Biomolecular Spectroscopy*, **2019**, 206, 642, <https://doi.org/10.1016/j.saa.2018.05.057>.
 20. Ganguly, R.; Sreenivasulu, B.; Vittal, J. J. *Coord. Chem. Rev.*, **2008**, 252, 1027, <https://doi.org/10.1016/j.ccr.2008.01.005>.
 21. Boghaei, D. M.; Gharagozlou, M. *Spectrochim. Acta Part A: Molecular and Biomolecular Spectroscopy*, **2007**, 944 <https://doi.org/10.1016/j.saa.2006.09.012>.
 22. Kobakhidze, N.; Farfán, N.; Romero, M.; Méndez-Stivalet, J. M.; Ballinas-López, M. G.; García-Ortega, H.; Domínguez, O.; Santillan, R.; Sánchez-Bartéz, F.; Gracia-Mora, I. *J. Organomet. Chem.*, **2010**, 695, 1189, <https://doi.org/10.1016/j.jorganchem.2010.01.024>.
 23. Lowe, J. T.; Panek, J. S. *Org. Lett.*, **2005**, 7, 1529 <https://doi.org/10.1021/ol0501875>.
 24. Lowe, J. T.; Panek, J. S. *Org. Lett.*, **2008**, 10, 3813 <https://doi.org/10.1021/ol801499s>.
 25. Lukesh, J. M.; Donaldson, W. A. *Tetrahedron Lett.*, **2005**, 46, 5529, <https://doi.org/10.1016/j.tetlet.2005.06.040>.
 26. Berberich, S. M.; Cherney, R. J.; Colucci, J.; Courillon, C.; Geraci, L. S.; Kirkland, T. A.; Marx, M. A.; Schneider, M. F.; Martin, S. F. *Tetrahedron*, **2003**, 59, 6819 [https://doi.org/10.1016/S0040-4020\(03\)00370-3](https://doi.org/10.1016/S0040-4020(03)00370-3).
 27. Dakin, L. A.; Panek, J. S. *Org. Lett.*, **2003**, 5, 3995, <https://doi.org/10.1021/ol035581m>.
 28. Su, Q.; Dakin, L. A.; Panek, J. S. *J. Org. Chem.*, **2007**, 72, 2, <https://doi.org/10.1021/jo0610412>.

29. Yadav, J. S.; Pattanayak, M. R.; Das, P. P.; Mohapatra, D. K. *Org. Lett.*, **2011**, *13*, 1710, <https://doi.org/10.1021/ol200223q>.
30. Raghavan, S.; Samanta, P. K. *Org. Lett.*, **2012**, *14*, 2346, <https://doi.org/10.1021/ol3007698>.
31. Lucas, B. S.; Luther, L. M.; Burke, S. D. *J. Org. Chem.*, **2005**, *70*, 3757, <https://doi.org/10.1021/jo050034v>.
32. Wang, B.; Hansen, T. M.; Wang, T.; Wu, D.; Weyer, L.; Ying, L.; Engler, M. M.; Sanville, M.; Leitheiser, C.; Christmann, M.; Lu, Y.; Chen, J.; Zunker, N.; Cink, R. D.; Ahmed, F.; Lee, C.-S.; Forsyth, C. J. *J. Am. Chem. Soc.*, **2011**, *133*, 1484, <https://doi.org/10.1021/ja108906e>.
33. Clarke, P. A.; Santos, S.; Mistry, N.; Burroughs, L.; Humphries, A. C. *Org. Lett.*, **2011**, *13*, 624, <https://doi.org/10.1021/ol102860r>.
34. Kumaraswamy, G.; Ramakrishna, G.; Naresh, P.; Jagadeesh, B.; Sridhar, B. *J. Org. Chem.*, **2009**, *74*, 8468, <https://doi.org/10.1021/jo901739y>.
35. Reddy, C. R.; Reddy, G. B.; Srikanth, B. *Tetrahedron: Asymmetry*, **2011**, *22*, 1725, <https://doi.org/10.1016/j.tetasy.2011.10.006>.
36. Gollner, A.; Altmann, K.-H.; Gertsch, J.; Mulzer, J. *Tetrahedron Lett.*, **2009**, *50*, 5790, <https://doi.org/10.1016/j.tetlet.2009.07.122>.
37. Paterson, I.; Menche, D.; Håkansson, A. E.; Longstaff, A.; Wong, D.; Barasoain, I.; Buey, R. M.; Díaz, J. F. *Bioorg. Med. Chem. Lett.*, **2005**, *15*, 2243, <https://doi.org/10.1016/j.bmcl.2005.03.018>.
38. Pânzariu A., Vasincu I.M., Dragostin O.M., Drăgan M., Buron F., Routier S., Profire L. *Farmacia*, **2015**, *63*, 4.
39. Barkova, N. P.; Keiko, N. A.; Stepanova, L. G.; Voronkov, M. G.; Portyanoi, A. A.; Nikiforova, T. I. Russian Patent no. 1 727 243, 1991; Byull. Izobret., **1993**, no. 33.
40. Chernyshev, K. A.; Krivdin, L. B.; Larina, L. I.; Konkova, T. V.; Demina, M. M.; Medvedeva, A. S. *Magn. Reson. Chem.*, **2007**, *45*, 661, <https://doi.org/10.1002/mrc.2017>.

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