2,2-Dimethyl-3-[(4-methylphenyl)sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole: synthesis, properties, and structure

A. Yu. Nikonov, I. V. Sterkhova, and N. F. Lazareva★

A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences, 1 ul. Favorskogo, 664033 Irkutsk, Russian Federation.
E-mail: nataly_lazareva@irioch.irk.ru

2,2-Dimethyl-3-[(4-methylphenyl)sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole was synthesized by the reaction of *N*-(2-hydroxyphenyl)-4-methylbenzenesulfonamide with dimethyldichlorosilane or *N*,*N*-bis(dimethylamino)dimethylsilane in 68 and 96% yield, respectively. The structure of the new compound was assigned using NMR and IR spectroscopy and confirmed by X-ray diffraction. In the crystal, the silole molecules are linked to each other by short O···H—C contacts (~2.6 Å) between the oxygen atoms of the bicyclic moiety and the methyl hydrogen atoms of adjacent molecules.

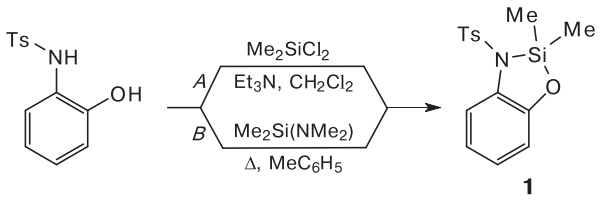
Key words: *N*-(2-hydroxyphenyl)-4-methylbenzenesulfonamide, silylation, 2,2-dimethyl-3-[(4-methylphenyl)sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole, hydrolysis, alcoholysis, X-ray diffraction.

Sulfonic acid amides $RSO_2NR'R$ (R, R′, R″ = Alk, Ar, Hetaryl) are widely used in synthetic organic and medicinal chemistry.[1—6] *para*-Aminobenzenesulfonamide derivatives have been commonly applied in medical practice as antibacterial agents since the mid-20th century. Investigations showed that compounds containing the SO_2N group exhibit a wide range of biological activities. In particular, these compounds possess antibacterial, antiviral, antifungal, anti-inflammatory, antituberculosis, antimalarial, antiprotozoal, and anticoagulant properties.[7—16] In recent years, sulfonic acid amides were found to have antitumor activity due to their ability to inhibit tubulin polymerization.[17—19] The biological activity of sulfonamides is largely determined by their ability to form non-covalent bonds (hydrogen and halogen bonds, complexation with metal sites of proteases, π-bonding with aromatic donors, *etc.*).[20—23]

The silyl modification of organic compounds is widely used in synthetic, medicinal, and analytical chemistry. The introduction of trialkylsilyl groups R_3Si as efficient, easily removable protecting groups has received great attention to series of O- and N-nucleophiles.[24—27] The Si/C bioisosterism,[28] which is of both theoretical interest and practical importance for the development of medicinal chemistry, is less known. It is worth noting that the bioisosteric replacement of the carbon moiety in the molecule with a silicon-containing group can lead to the enhancement of the therapeutic effect and a decrease in toxicity of the compound.[29—31] Silicon-containing sulfonamides are poorly studied. Recently, we synthesized *N*-{[2-(trimethylsilyl)oxy]-phenyl}-4-methylphenyl- and *N*-{[2-(trimethylsilyl)oxy]-phenyl}-4-chlorophenylsulfonamides and investigated their structures.[32] As part of our continuing research, we synthesized 2,2-dimethyl-3-[(4-methylphenyl)sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole (**1**), determined its structure, and investigated selected properties.

N-(2-Hydroxyphenyl)-4-methylbenzenesulfonamide reacts with dimethyldichlorosilane in dichloromethane at room temperature in the presence of triethylamine as a hydrogen chloride acceptor (Scheme 1) to form 2,2-dimethyl-3-[(4-methylphenyl)sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole (**1**) in 68% yield (method *A*).

Scheme 1



1

Variations of the experimental conditions (the solvent and the hydrogen chloride acceptor) did not lead to an increase in the yield of the target product. The main problem is that the largest loss of the product occurs in the step of its isolation from the reaction mixture. An alternative method of synthesis (method *B*) is more

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 2, pp. 386—390, February, 2021.

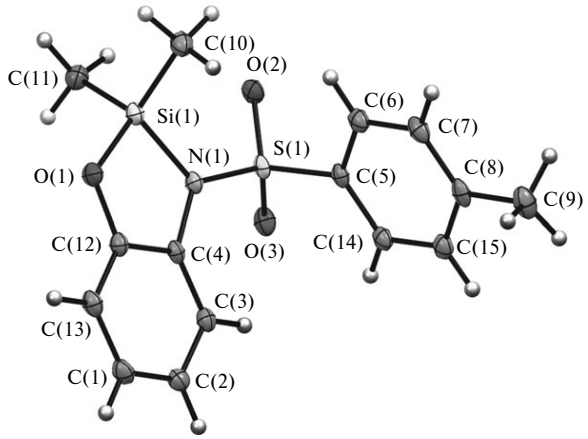
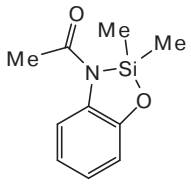


Fig. 1. Molecular structure of compound **1** with displacement ellipsoids drawn at the 50% probability level.

efficient and allows the preparation of compound **1** in nearly quantitative yield (96%). This method is based on the reaction of *N*-(2-hydroxyphenyl)-4-methylbenzenesulfonamide with *N*,*N*-bis(dimethylamino)dimethylsilane (see Scheme 1). The reaction occurs at reflux in toluene within 6—8 h. The completion of the reaction is evidenced by the cessation of the release of dimethylamine from the reaction mixture (litmus paper).

The structure of compound **1** was assigned using IR and NMR spectroscopy and confirmed by X-ray diffraction. The molecular structure of compound **1** is shown in Fig. 1. Selected structural parameters are given in Table 1. There is one molecule of compound **1** per asymmetric unit. No structural analogs of compound **1** are avaialable in the Cambridge Crystallographic Data Centre. 3-Acetyl-2,2-dimethyl-2,3-dihydro-1,3,2-benzoxazasilole (**2**), which was synthesized in our recent research,[33] is structurally most similar to compound **1**.

Compounds **1** and **2** have very similar geometric parameters of the five-membered heterocycle. The Si—O and Si—N distances in the heterocycle of compound **1** are 1.683 and 1.789 Å, respectively. The O—Si—N, Si—N—C, and C—O—Si bond angles are 91.18°, 110.49°, and 113.11°, respectively. The differences in the geometric parameters of compounds **1** and **2** are at most 0.06 Å and 0.5°. In the crystal, the molecules of compound **1** are linked to each other by short CH···O contacts (2.638 and 2.681 Å) between the hydrogen atoms of $SiMe_2$ groups and the oxygen atoms of adjacent molecules (Fig. 2).



2

Compound **1** is extremely hygroscopic and is easily hydrolyzed by atmospheric moisture. The mild hydrolysis of this compound with atmospheric moisture leads to the opening of the heterocycle at the Si—N bond to form the corresponding silanole, *N*-[2-(hydroxydimethylsilyloxy)phenyl]-4-methylphenylsulfonamide (**3**) (Scheme 2).

The resulting silanole is a yellowish powdered compound soluble in aromatic hydrocarbons, chloroform, and acetonitrile. Unfortunately, we failed to obtain crystals of compound **3** suitable for X-ray diffraction. Both the hydrolysis of compound **1** in a solvent using an equimolar amount of water and the storage of the solid sample within a week lead to the decomposition of silanole **3** into *N*-(2-hydroxyphenyl)-4-methylphenylsulfonamide and polysiloxanes of unidentified structure. The storage of a solution of compound **3** in acetonitrile above molecular sieves 4 Å resulted in the formation of *N*,*N*′-[(1,1,3,3-tetramethyldisiloxane-1,3-diyl)bis(oxy-2,1-phenylene]bis(4-methylphenylsulfonamide) (**4**) as the condensation product (see Scheme 2).

The reaction of compound **1** with *tert*-butanol was accompanied by the Si—N bond cleavage and gave the corresponding *N*-{2-[*tert*-butoxy(dimethyl)silyloxy]phenyl}-4-methylphenylsulfonamide **5** (see Scheme 2).

Table 1. Selected bond lengths (*d*), bond angles (ω), and torsion angles (φ) in molecule **1**

Bond	*d*/Å	Bond angle	ω/deg	Torsion angle	φ/deg
Si(1)—O(1)	1.683(1)	O(1)—Si(1)—N(1)	91.2(1)	N(1)—Si(1)—O(1)—C(12)	−1.3(1)
Si(1)—N(1)	1.789(1)	O(1)—Si(1)—C(10)	111.2(1)	C(10)—Si(1)—O(1)—C(12)	−117.7(1)
Si(1)—C(10)	1.839(2)	N(1)—Si(1)—C(10)	113.9(1)	O(3)—S(1)—N(1)—C(4)	46.6(1)
Si(1)—C(11)	1.839(2)	O(1)—Si(1)—C(11)	113.6 (1)	C(11)—Si(1)—O(1)—C(12)	111.7(1)
S(1)—O(2)	1.442(1)	N(1)—Si(1)—C(11)	110.5(1)	O(2)—S(1)—N(1)—C(4)	175.6(1)
S(1)—O(3)	1.438 (1)	C(10)—Si(1)—C(11)	114.3(1)	C(5)—S(1)—N(1)—C(4)	−69.8(1)
S(1)—N(1)	1.629(1)	C(12)—O(1)—Si(1)	113.1(1)	O(3)—S(1)—N(1)—Si(1)	−138.3(1)
S(1)—C(5)	1.761(1)	O(3)—S(1)—O(2)	119.7(1)	O(2)—S(1)—N(1)—Si(1)	−9.2(1)
O(1)—C(12)	1.377(2)	O(2)—S(1)—N(1)	103.7(1)	C(5)—S(1)—N(1)—Si(1)	105.4(1)
N(1)—C(4)	1.424(2)	O(3)—S(1)—N(1)	109.9(1)	O(1)—Si(1)—N(1)—C(4)	2.0(1)
C(1)—C(2)	1.390(2)	O(3)—S(1)—C(5)	108.2(1)	C(10)—Si(1)—N(1)—C(4)	115.9(1)
C(2)—C(3)	1.402(2)	O(2)—S(1)—C(5)	109.0(1)	C(1)—C(2)—C(3)—C(4)	−0.4(2)
C(3)—C(4)	1.387(2)	N(1)—S(1)—C(5)	105.5(1)	C(11)—Si(1)—N(1)—C(4)	−113.8(1)
C(8)—C(9)	1.505(2)	C(4)—N(1)—S(1)	125.3(1)	O(1)—Si(1)—N(1)—S(1)	−173.8(1)

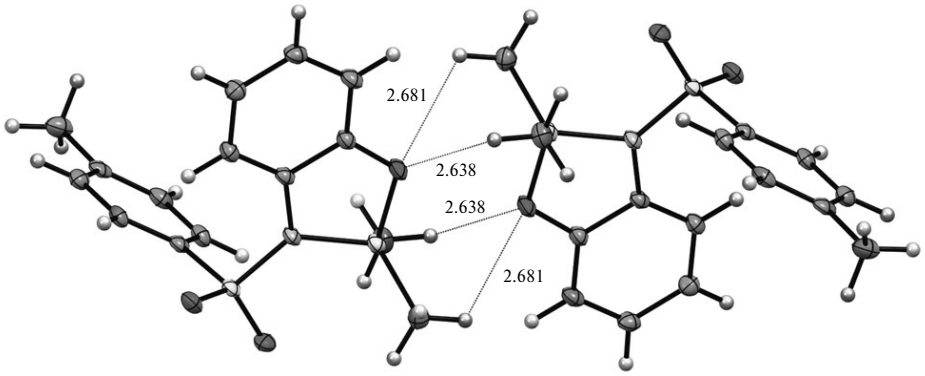
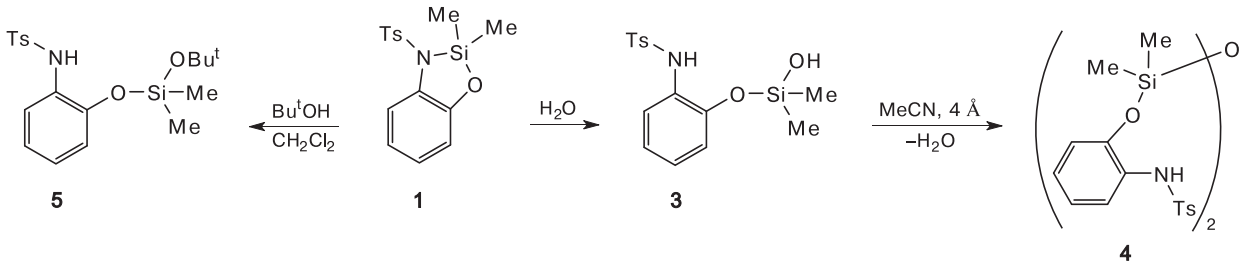


Fig. 2. Short intermolecular contacts in the crystal of compound **1**.

Scheme 2



To conclude, the reaction of *N*-(2-hydroxyphenyl)-4-methylbenzenesulfonamide with *N*,*N*-bis(dimethylamino)-dimethylsilane allows the synthesis of 2,2-dimethyl-3-[(4-methylphenyl)sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole (**1**) in nearly quantitative yield (96%). This reaction with dimethyldichlorosilane affords the target product in much lower yield (68%). The structure of 2,2-dimethyl-3-[(4-methylphenyl)sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole was confirmed by NMR and IR spectroscopy and X-ray diffraction. The further investigation of the properties of polyfunctional 2,2-dimethyl-3-[(4-methylphenyl)-sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole and its analogs holds promise for the following reasons. First, compounds containing a Si—N bond are commonly applied in organic synthesis[34—36] and materials science.[37,38] Second, compounds containing the sulfonamide group exhibit a wide range of biological activities.[7—16]

Experimental

The NMR spectra were recorded on a Bruker DPX-400 spectrometer (^{1}H, 400.1 MHz; ^{13}C, 100.6 MHz; ^{29}Si, 79.5 MHz) in $CDCl_3$ using HMDS and cyclohexane as the internal standard. The IR spectra of amide **1** were measured as KBr pellets on a Varian 3100 FT-IR spectrophotometer. A single crystal of compound **1** was obtained by the crystallization from a benzene—hexane mixture (5 : 1). The X-ray diffraction data were collected on a Bruker D8 Venture diffractometer (Mo-Kα radiation, λ = 0.71073 Å) using the φ- and ω-scanning technique. The structure was solved by direct methods using the SHELX program package.[39] An absorption correction was applied with the SADABS program. All nonhydrogen atoms were refined with anisotropic displacement parameters using the SHELX program package.[39] Crystallographic data for molecule **1** were deposited with the Cambridge Crystallographic Data Centre (www.ccdc.cam.ac.uk/data_request/cif, CCDC 1947409).

The X-ray crystallographic data for compound **1**: $C_{15}H_{17}NO_3SSi$, M = 319.44, colorless prismatic crystals, 0.40×0.50×0.50 mm. The crystal belongs to the monoclinic system, space group $P2_1/c$; $\theta_{min}/\theta_{max}$ = 2.67/30.21; T = 100 K, a = 12.642(2) Å, b = 8.743(2) Å, c = 13.729(3) Å; β = 90.38(1)°, V = 1517.3(5) Å^3, Z = 4, d_{calc} = 1.398 g cm^3, F(000) = 760; the absorption coefficient μ = 0.301 mm^{-1}; a total of 66798 reflections, of which 4459 reflections were unique; 193 refined parameters; R = 4.43, R_w = 0.052 (for all reflections); goodness of fit based on F^2 is 1.047; $\Delta\rho_{max}/\Delta\rho_{min}$ = 0.708/−0.851 e Å^{-3}; the weighting scheme $w = [\sigma^2 (F_0^2) + (0.0392P)^2 + 1.3952P]^{-1}$, where $P = (F_0^2 + 2Fc^2)/3$.

N-(2-Hydroxyphenyl)-4-methylphenylsulfonamide was synthesized by a procedure described in our previous study.[33] *N*,*N*-Bis(dimethylamino)dimethylsilane was synthesized by a method described in the literature.[40] The solvents and reagents were prepared by standard procedures[41] before use.

2,2-Dimethyl-3-[(4-methylphenyl)sulfonyl]-2,3-dihydro-1,3,2-benzoxazasilole (1). Method *A*. A solution of Me_2SiCl_2 (1.29 g, 0.01 mol) in CH_2Cl_2 (15 mL) was slowly added dropwise to a vigorously stirred solution of *N*-(2-hydroxyphenyl)-4-methylphenylsulfonamide (2.63 g, 0.01 mol) in dichloromethane (150 mL) in the presence of triethylamine (5 g, excess). The

resulting reaction mixture was heated with stirring on a water bath at 60 °C for 18 h. The solvent was removed from the reaction mixture on a rotary evaporator, and then benzene (50 mL) was added. After the cooling, the precipitate of triethylamine hydrochloride that formed was filtered off, washed with benzene, and combined with the filtrate. The filtrate was concentrated to dryness. According to the NMR spectrum, the resulting product was a mixture of compound **1** and triethylamine hydrochloride. Compound **1** was isolated by the recrystallization from a benzene—hexane mixture (5 : 1). The yield was 2.17 g (68%), compound **1** decomposes at a temperature above 152 °C. ^{1}H NMR ($CDCl_3$), δ: 0.75 (s, 6 H, Me_2Si); 2.39 (s, 3 H, Me); 6.69—7.86 (m, 8 H, H_{Ar}). ^{13}C NMR, δ: 0.92 (Me_2Si); 26.99 (Me); 113.79, 121.06, 123.10, 127.25, 127.44, 129.58, 129.92, 136.69, 144.31, 149.15 (2 Ar). ^{29}Si NMR ($CDCl_3$), δ: 25.89. Found (%): C, 56.21; H, 5.19; N, 4.52. $C_{15}H_{17}NO_3SSi$. Calculated (%): C, 56.40; H, 5.36; N, 4.38.

Method *B*. A mixture of *N*-(2-hydroxyphenyl)-4-methylbenzenesulfonamide (2.63 g, 0.01 mol) and *N*,*N*-bis(dimethylamino)dimethylsilane (1.46 g) in toluene (50 mL) was heated until dimethylamine ceased to be released (litmus paper). The solution was concentrated to 4/5 of the initial volume. The crystals that formed were washed with pentane and dried *in vacuo*. The yield was 3.07 g (96%). The spectral characteristics are identical to those reported above.

***N*-(2-{[Hydroxy(dimethyl)silyl]oxy}phenyl)-4-methylphenylsulfonamide (3).** Compound **1** (0.32 g, 0.001 mol) was kept in an open vessel for 36 h at room temperature, after which a yellowish amorphous powder was obtained. The product was washed with cold diethyl ether and dried *in vacuo*. The yield was 0.34 g (~100%), compound **3** decomposes at 196 °C. ^{1}H NMR, δ: 0.37 (s, 6 H, Me_2Si); 2.39 (s, 3 H, Me); 6.92—7.69 (m, 8 H, H_{Ar}); 5.92 (br.s, 1 H, NH); 8.34 (br.s, 1 H, SiOH). ^{13}C NMR, δ: 1.32 (Me_2Si); 26.82 (Me); 113.56, 119.84, 122.97, 127.46, 127.75, 129.64, 130.12, 136.78, 144.62, 149.59 (2 Ar). ^{29}Si NMR, δ: −9.72. Found (%): C, 53.62; H, 5.27; N, 4.35. $C_{15}H_{19}NO_4SSi$. Calculated (%): C, 53.39; H, 5.68; N, 4.15.

***N*,*N*′-[(1,1,3,3-Tetramethyldisiloxane-1,3-diyl)bis(oxy-2,1-phenylene)]bis(4-methylphenylsulfonamide) (4).** Molecular sieves 4 Å (1 g) were added to a solution of compound **3** (0.32 g, 0.001 mol) in acetone (25 mL). The mixture was stirred for 48 h at room temperature. Then the solvent was removed. Attempts to crystallize the resulting viscous product failed. The yield was 0.31 g (~100%). ^{1}H NMR, δ: 0.39 (s, 12 H, Me_2Si); 2.40 (s, 6 H, Me); 6.24 (br.s, 2 H, 2 NH); 6.90—7.74 (m, 16 H, H_{Ar}). ^{13}C NMR, δ: 2.56 (Me_2Si); 26.86 (Me); 113.58, 119.92, 123.04, 127.48, 127.86, 129.72, 130.25, 136.79, 144.68, 150.12 (4 Ar). ^{29}Si NMR, δ: 7.84. Found (%): C, 54.93; H, 5.65; N, 4.47. $C_{30}H_{36}N_2O_7S_2Si_2$. Calculated (%): C, 54.85; H, 5.52; N, 4.26.

***N*-{2-[*tert*-Butoxy(dimethyl)silyloxy]phenyl}-4-methylphenylsulfonamide (5).** A solution of *tert*-butanol (0.15 g, 0.002 mol) in CH_2Cl_2 (5 mL) was added dropwise to a solution of compound **1** (0.64 g, 0.002 mol) in CH_2Cl_2 (15 mL). The reaction mixture was refluxed for 4 h. The solvent was removed *in vacuo*, and the residue (opalescent viscous substance) was stored *in vacuo* for 3 h and then analyzed. The yield was 0.78 g (~100%). ^{1}H NMR, δ: 0.32 (s, 6 H, Me_2Si); 1.27 (s, 9 H, Me_3C); 2.40 (s, 3 H, Me); 4.32 (br.s, 1 H, NH); 6.74—7.69 (m, 8 H, H_{Ar}). ^{13}C NMR, δ: −1.56 (Me_2Si); 26.58 (Me); 32.37 ($\underline{Me}_3C$); 78.64 ($\underline{C}$—Me_3); 113.70, 119.65, 122.56, 127.39, 127.72, 129.40, 130.08, 136.56, 144.49, 149.42 (2 Ar). ^{29}Si NMR, δ: −6.96. Found (%): C, 57.83; H, 6.86; N, 3.48. $C_{19}H_{27}NO_4SSi$. Calculated (%): C, 57.98; H, 6.92; N, 3.56.

This work was financially supported by the Russian Foundation for Basic Research (Project No. 18-33-00368-mol_a). The main results were obtained using equipment of the Baikal Analytical Center for Collective Use of the Siberian Branch of the Russian Academy of Sciences.

References

1. S. Bhadra, H. Yamamoto, *Chem. Rev.*, 2018, **118**, 3391; DOI: 10.1021/acs.chemrev.7b00514.
2. C. Sambiagio, D. Schönbauer, R. Blieck, T. Dao-Huy, G. Pototschnig, P. Schaaf, T. Wiesinger, M. F. Zia, J. Wencel-Delord, T. Besset, B. U. W. Maes, M. Schnürch, *Chem. Soc. Rev.*, 2018, **47**, 6603; DOI: 10.1039/c8cs00201k.
3. P. Ribiere, V. Declerck, J. Martinez, F. Lamaty, *Chem. Rev.*, 2006, **106**, 2249; DOI: 10.1021/cr0300587.
4. J. D. Wilden, *J. Chem. Res.*, 2010, **34**, 541; DOI: 10.3184/030823410X12857514635822.
5. J. A. Halfen, *Curr. Org. Chem.*, 2005, **9**, 657; DOI: 10.2174/1385272053765024.
6. H. Yang, R. G. Carter, *Synlett.*, 2010, 2827; DOI: 10.1055/s-0030-1259020.
7. F. Carta, C. T. Supuran, A. Scozzafava, *Future Med. Chem.*, 2014, **6**, 1149; DOI: 10.4155/fmc.14.68.
8. T. V. Wani, S. Bua, P. S. Khude, A. H. Chowdhary, C. T. Supuran, M. P. Toraskar, *J. Enzym. Inhib. Med. Chem.*, 2018, **33**, 962; DOI: 10.1080/14756366.2018.1471475.
9. S. S. A. Shah, G. Rivera, M. Ashfaq, *Mini Rev. Med. Chem.*, 2013, **13**, 70; DOI: 10.2174/1389557511307010070.
10. R. Kh. Bagautdinova, L. I. Vagapova, A. V. Smolobochkin, A. S. Gazizov, A. R. Burilov, M. A. Pudovik, A. D. Voloshina, *Mendeleev Commun.*, 2019, **29**, 686; DOI: 10.1016/j.mencom.2019.11.028.
11. I. Melnikova, *Nat. Rev. Drug Discov.*, 2005, **4**, 453; DOI: 10.1038/nrd1755.
12. A. Bertolini, A. Ottani, M. Sandrini, *Curr. Med. Chem.*, 2002, **9**, 1033; DOI: 10.2174/0929867024606650.
13. J. Dong, Y. Wang, Q. Xiang, X. Lv, W. Weng, Q. Zeng, *Adv. Synth. Catal.*, 2013, **355**, 692; DOI: /10.1002/adsc.201200752.
14. D.-W. Zhang, X. Zhao, J.-L. Hou, Z.-T. Li, *Chem. Rev.*, 2012, **112**, 5271; DOI: 10.1021/cr300116k.
15. N. N. Farshori, A. Ahmad, A. U. Khan, A. Rauf, *Eur. J. Med. Chem.*, 2011, **46**, 1433; DOI: 10.1016/j.ejmech.2011.01.070.
16. P. Panini, R. G. Gonnade, D. Chopra, *New J. Chem.*, 2016, **40**, 4981; DOI: 10.1039/C5NJ03211C.
17. H.-Y. Lee, S.-L. Pan, M.-C. Su, Y.-M. Liu, C.-C. Kuo, Y.-T. Chang, J.-S. Wu, C.-Y. Nien, S. Mehndiratta, C.-Y. Chang, S.-Y. Wu, M.-J. Lai, J.-Y. Chang, J.-P. Liou, *J. Med. Chem.*, 2013, **56**, 8008; DOI: 10.1021/jm4011115−8018.
18. J. Kim, P. Chun, H. R. Moon, *Bull. Korean Chem. Soc.*, 2013, **34**, 1487; DOI:10.5012/BKCS.2013.34.5.1487.
19. R. Mahesh, V. L. Nayak, K. S. Babu, S. Riyaz, T. B. Shaik, G. B. Kumar, P. L. Mallipeddi, C. R. Reddy, K. C. Shekar, J. Jose, N. Nagesh, A. Kamal, *Chem. Med. Chem.*, 2017, **12**, 678; DOI: 10.1002/cmdc.201600643.

20. J. Mortier, C. Rakers, M. Bermudez, M. S. Murgueitio, S. Riniker, G. Wolber, *Drug Discov. Today*, 2015, **20**, 686; DOI: 10.1016/j.drudis.2015.01.003.
21. H. Zhao, A. Caflisch, *Eur. J. Med. Chem.*, 2015, **91**, 4; DOI: 10.1016/j.ejmech.2014.08.004-14.
22. *Structure-Based Drug Discovery*, Ed. L. W. Tari, Springer, New York, 2012, 385 p.; DOI: 10.1007/978-1-61779-520-6.
23. *Noncovalent Forces*, Ed. S. Scheiner, Springer, Cham Heidelberg, 2015, 529 p.; DOI: 10.1007/978-3-319-14163-3.
24. E. Lukevics, S. Germane, I. Segal, A. Zablotskaya, *Chem. Heterocycl. Compd.*, 1997, **33**, 234; DOI: 10.1007/BF02256766.
25. M. V. Kashutina, S. L. Ioffe, V. A. Tartakovskii, *Russ. Chem. Rev.*, 1975, **44**, 1620; DOI: 10.1070/RC1975v044n09 ABEH002373.
26. M. D. Mizhiritskii, Yu. A. Yuzhelevskii, *Russ. Chem. Rev.*, 1987, **56**, 355; DOI: 10.1070/RC1987v056n04ABEH003276.
27. G. Look, *Silylating Agents,* Buchs, Fluka Chemie AG, 1995, 43 p.
28. G. A. Patani, E. J. LaVoie, *Chem. Rev.,* 1996, **96**, 3147; DOI: 10.1021/cr950066q.
29. N. A. Meanwell, *J. Med. Chem.*, 2011, **54**, 2529; DOI: 10.1021/jm1013693−2591.
30. J. S. Mills, G. A. Showell, *Expert Opin. Invest. Drugs*, 2004, **13**, 1149; DOI: 10.1517/13543784.13.9.1149.
31. A. K. Franz, S. O. Wilson, *J. Med. Chem.*, 2013, **56**, 388; DOI: 40510.1021/jm3010114.
32. A. Yu. Nikonov, I. V. Sterkhova, V. Yu. Serykh, N. A. Kolyvanov, N. F. Lazareva, *J. Mol. Struct.*, 2019, **1198**, 126782; DOI: 10.1016/j.molstruc.2019.07.029.
33. N. F. Lazareva, A. Yu. Nikonov, N. N. Chipanina, L. P. Oznobikhina, I. V. Sterkhova, A. I. Albanov, *J. Organomet. Chem.*, 2017, **846**, 88; DOI: 10.1016/j.jorganchem.2017.05.061.
34. Y. Tanabe, T. Misaki, M. Kurihara, A. Iida, Y. Nishii, *Chem. Commun.*, 2002, 1628; DOI: 10.1039/b203783c.
35. B. Minkovich, I. Ruderfer, A. Kaushansky, D. Bravo-Zhivotovskii, Y. Apeloig, *Angew. Chem., Int. Ed.*, 2018, **57**, 13261; DOI: 10.1002/anie.201807027.
36. E. K. J. Lui, J. W. Brandt, L. L. Schafer, *J. Am. Chem. Soc.,* 2018, **140,** 4973; DOI: 10.1021/jacs.7b13783.
37. G. Glatz, T. Schmalz, T. Kraus, F. Haarmann, G. Motz, R. Kempe, *Chem. Eur. J.,* 2010, **16**, 4231; DOI: 10.1002/chem.200902836.
38. E. Kroke, Y.-L. Li, C. Konetschny, E. Lecomte, C. Fasel, R. Riedel, *Mater. Sci. (Engl.) R: Reports*, 2000, **26**, 97; DOI: 10.1016/S0927-796X(00)00008-5.
39. G. M. Sheldrick, *Acta Crystallogr.*, 2008, **D64**, 112; DOI: 10.1107/S010876730704393.
40. V. Passarelli, F. Benetollo, P. Zanella, G. Carta, G. Rossetto, *Dalton Trans.*, 2003, 1411; DOI: 10.1039/B212705A.
41. W. L. F. Armarego, C. L. L. Chai, *Purification of Laboratory Chemicals*, 6th Ed., Elsevier, 2009, 760 c.

Received June 23, 2020;
in revised form October 23, 2020;
accepted October 27, 2020