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An Efficient and Convenient Synthesis of 2-Thio[1,2,4]triazolo[1,5-*c*]quinazoline and its Derivatives

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Summary. 4-Hydrazinoquinazoline with carbon disulfide underwent a recyclization reaction. The title compound 2-thio[1,2,4]triazolo[1,5-*c*]quinazoline was obtained after treatment of 4-hydrazinoquinazoline with potassium ethylxanthogenate *via* a facile in situ *Dimroth*-like rearrangement of the expected [4,3-*c*] system. Its structure was established by X-ray diffraction study and confirmed by an independent synthesis, starting from *o*-aminobenzonitrile.

Keywords. Crystallographic structure analysis; Cyclization; 4-Hydrazinoquinazoline; Rearrangement; 2-Thio[1,2,4]triazolo[1,5-*c*]quinazoline.

Introduction

An increasing interest in S-substituted 2-thio[1,2,4]triazolo[1,5-*c*]quinazolines is caused by their pharmacological properties. Recently 5-substituted [1,2,4]triazolo[1,5-*c*]quinazolin-2-thiones have been found to exhibit antibacterial [1–3] and surface activity [2, 3]. There are some known synthesis approaches to these derivatives. The first one is an interaction of proper 4-oxo-4*H*-benzo[*a*][1,3]oxazines with thiosemicarbazide in pyridine [4, 5], glacial acetic acid [1, 6], or by fusion [2]. The second one includes the reaction of 3-amino-2-methyl-3*H*-quinazolin-4-one with thiourea [1], and also thermolysis of 2-substituted *N*-[4-oxo-3(4*H*)-quinazolinyl]thioureas, which yields the 5-substituted [1,2,4]triazolo[1,5-*c*] quinazolin-2-thiones [1, 3].

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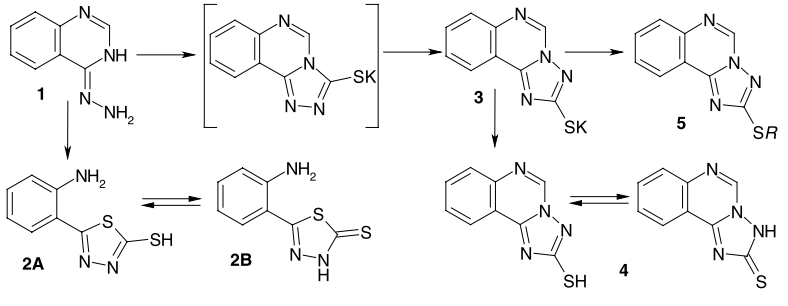
Since the synthesis of 2-thio[1,2,4]triazolo[1,5-*c*]quinazoline and its derivatives starting from 4-hydrazinoquinazoline were hitherto unknown, our efforts were directed towards the development of new preparative methods of synthesis of the title derivatives.

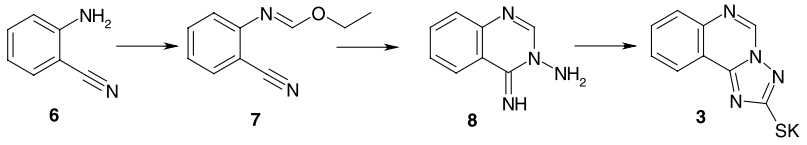
An extremely facile rearrangement of the [1,2,4]triazolo[4,3-*c*]quinazolines to the isomeric [1,5-*c*] series [7, 8] can be used for the one-pot synthesis of 2-thio[1,2,4]triazolo[1,5-*c*]quinazolines. The known method of a quinazoline ring annulation to the 5-thioxo-1,2,4-triazole based on the reaction of substituted 4-hydrazinoquinazolines with carbon disulfide, which is accompanied by the formation of proper 3-thio[1,2,4]triazolo[4,3-*c*]quinazolines [9–11]. Reaction of an unsubstituted 4-hydrazinoquinazoline with carbon disulfide in chloroform was reported to form [1,2,4]triazolo[4,3-*c*]quinazolin-3-thiole [12]. However, *Coppola* and *Hardtmann* observed, that the expected compound in the presence of ethanolic potassium hydroxide was not formed. Instead, 2-(2-aminophenyl)-5-mercapto-1,3,4-thiadiazole was isolated as indicated by the physical and spectral data, and its structure was confirmed by independent synthesis [13].

Results and Discussion

It is of interest to verify whether 4-hydrazinoquinazoline (**1**) undergoes the recyclization reaction under the conditions mentioned above [13] as well as in chloroform [12], 2-propanol, or dioxane without base. As a result, a compound was obtained whose physical constants were identical with those of 2-(2-aminophenyl)-5-mercapto-1,3,4-thiadiazole (**2**) in all cases (Scheme 1). Thus, the disappearance of the singlet of H-2 and a low-field shift of the aromatic protons as well as a broadened two-proton singlet of NH_2-group in the ^{1}H NMR spectrum of **2** indicates the pyrimidine ring opening. Furthermore, a broad one-proton singlet at $\delta = 14.62$ ppm was observed, which belongs to the NH-proton. Thus, the latter compound exists as the thioxo-tautomer, without any detectable quantity of the SH-tautomeric form in *DMSO*-d_6 solution. Hence, the structure of the obtained compound should be assigned to the 5-(2-aminophenyl)-1,3,4-thiadiazol-2(3*H*)-thione (**2B**). A predominance of the thioxo tautomeric form was observed also for other related compounds [14]. Finally, the chromatomass spectrum (APCI) of **2** was found to show the peak of protonated molecular ion $[MH]^+$ at $m/z = 210$



Scheme 1



Scheme 2

(100%, base peak). Basic directions of its fragmentation are related to the elimination of sulfur $[MH–S]^+$ ($m/z = 178$), and SCN from the thiadiazole cycle $[MH–SCN]^+$ ($m/z = 152$), that additionally confirms the structure of **2**. The possible mechanism of the indicated recyclisation has been discussed in Ref. [13].

In this respect, **1** differs in its reaction with carbon disulfide from those of several others substituted in 2 position. Further investigation was directed towards the synthesis of the target *s*-triazoloquinazoline system. When **1** was allowed to react with potassium ethylxanthogenate in 2-propanol, potassium 2-thio[1,2,4]triazolo[1,5-*c*]quinazoline (**3**) was isolated as a sole product. The structure of **3** was confirmed by independent synthesis starting from *o*-aminonitrile **6**, which is known as a very useful and versatile substrate for pyrimidine ring construction [15]. Condensation of *o*-aminobenzonitrile (**6**) with triethyl orthoformate afforded the intermediate ethoxymethyleneamino derivative **7** (Scheme 2), which was used without purification in the next step. Treatment of **7** with hydrazine hydrate in ethanol yielded 4-imino-3(4*H*)-quinazolinamine (**8**). Cyclocondensation of **8** with potassium ethylxanthogenate in 2-propanol resulted in the formation of the same potassium 2-thio[1,2,4]triazolo[1,5-*c*]quinazoline (**3**) (Scheme 2).

Thus, we have found that reaction of **1** with K ethylxanthogenate yielded **3** by a facile *in situ Dimroth*-like rearrangement of the [4,3-*c*] system (Scheme 1). The potassium salt **3** was converted to the target 2-thio-[1,2,4]triazolo[1,5-*c*]quinazoline (**4**) with dilute HCl. The latter compound **4** in *DMSO*-d_6 solution can exist as one of the tautomeric forms as well as their equilibrium mixture. When potassium salt **3** was treated with alkylhalides, phenacyl chloride, and chloroacetic acid under mild conditions, the alkylation was found to proceed smoothly at the sulfur atom resulting in 2-(alkylthio)-[1,2,4]triazolo[1,5-*c*]quinazolines (**5a**–**5e**) (Scheme 1).

The possibility of mistaking the structures obtained cannot be denied. Thus, in order to confirm the structure of the *s*-triazoloquinazolines **3**–**5** unambiguously and to remove any doubt, a single crystal X-ray diffraction study of **5b** was carried out (Fig. 1). According to the crystallographic data, all non-hydrogen atoms of the

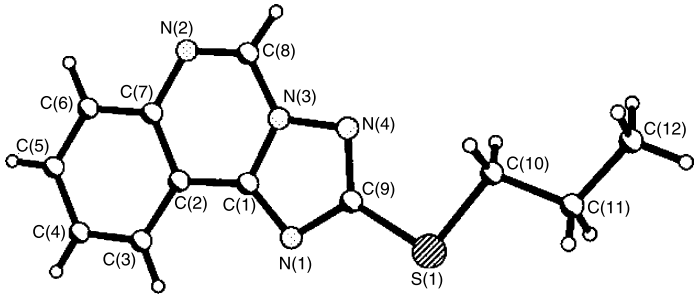


Fig. 1. X-ray molecular structure of **5b** with the atom numbering used in crystallographic analysis

molecule **5b** are co-planar within 0.01 Å. The propyl substituent has *syn*-orientation with respect to the N(4)–C(9) bond (the C(10)–S(1)–C(9)–N(4) torsion angle is −0.6(1)°) and adopts a *trans–trans* conformation (the C(9)–S(1)–C(10)–C(11) and S(1)–C(10)–C(11)–C(12) torsion angles are −176.35(9) and −178.29(9)°, respectively). In the crystal, molecules **5b** form stacks along the crystallographic direction [100], bounded by the very week intermolecular hydrogen bond C(8)–H(8) ··· N(4)′ (1-*x*, -*y*, 1-*z*, H ··· N 2.47 Å, C–H ··· N 162°).

In the mass spectrum (APCI) of **5a**–**5e** the base peaks of appropriately protonated molecular ions $[MH]^+$ were observed with 100% intensivity. The mass spectra (EI) of **5b**, **5d**, and **5e** were found to show molecular ion peaks whose fragmentation patterns were in agreement with their structures. The ^{1}H NMR spectra of **5a**–**5e** recorded in *DMSO*-d_6 showed signals from the *s*-triazoloquinazoline moiety and substituents. It is noteworthy, that the singlet of H-5 at 9.53–9.30 ppm shifted downfield as a result of the tricyclic system formation. The ^{13}C NMR spectra were also in good agreement with the assigned structures of **5b**, **5d**, and **5e**.

In conclusion, a useful and convenient method for 2-thio[1,2,4]triazolo[1,5-*c*]quinazoline (**4**) preparation has been worked out. The structure of intermediate potassium salt **3** was confirmed by an independent synthesis starting from *o*-aminobenzonitrile (**6**). Alkylation of **3** with alkylhalides, chloroacetic acid, and phenacyl chloride was found to proceed smoothly at the sulfur atom resulting in 2-(alkylthio)[1,2,4]triazolo[1,5-*c*]quinazolines **5a**–**5e**. Their structure was deduced from the X-ray diffraction study of **5b**. Moreover, **3**–**5** were prepared starting from the readily available **1**. This fact along with simple experimental technique used provides an easy access to the title derivatives.

Experimental

4-Hydrazinoquinazoline (**1**) was prepared as reported [16]. Other starting materials were commercially available and used without additional purification. All mps were determined in open capillary tubes in a *Thiele*'s apparatus. ^{1}H NMR spectra were recorded on a Mercury 400 (400 MHz) spectrometer with Si*Me*$_4$ as internal standard in *DMSO*-d_6 solution, if not otherwise noted. ^{13}C NMR spectra were recorded on a Bruker Avance 500 (125 MHz for ^{13}C) spectrometer in *DMSO*-d_6 solution. Mass spectra were determined on a Varian 1200 L instrument (EI, 70 eV). Chromatomass spectra were determined on an Agilent 1100 instrument (atmospheric pressure chemical ionization – APCI). The purity of all compounds prepared was checked by ^{1}H NMR and chromatomass spectra and for all compounds satisfactory elemental analyses were obtained.

5-(2-Aminophenyl)-1,3,4-thiadiazol-2(3H)-thione (**2B**)

To a solution of 1.6 g **1** (10 mmol) in 20 cm^3 *Et*OH were added 2 cm^3 CS_2 (33 mmol) and a solution of 0.62 g KOH (11 mmol) in 10 cm^3 H_2O. The resulting mixture was refluxed for 3 h. All insoluble materials were filtered off from the reaction mixture and the solvent was removed under reduced pressure. To the residue were added 30 cm^3 5% KOH solution and any insoluble material was filtered off. The resulting solution was neutralized with 50% aqueous acetic acid, and the yellow precipitate was filtered off and washed well with H_2O. Recrystallization from *Et*OH furnished 1.0 g (48%) **2B**. Mp 214–216°C (Ref. [13] 214–216°C); ^{1}H NMR: $\delta = 14.62$ (s, NH), 7.21 (m, H-4, H-6), 6.85 (d, $J = 7.8$ Hz, H-3), 6.62 (t, $J = 7.6$ Hz, H-5), 6.49 (br.s, NH_2) ppm; MS (APCI): $m/z = 210$ (MH^+), 209, 178, 179, 152, 150, 138, 118.

Potassium 2-thio[1,2,4]triazolo[1,5-c]quinazoline (**3**, $C_9H_5KN_4S$)
Method A. A mixture of 3.2 g **1** (20 mmol) and 3.2 g potassium ethyl-xanthogenate (20 mmol) in 40 cm^3 2-propanol was refluxed for 4 h. After cooling, the precipitate was filtered off, washed with 2-propanol and acetone, and dried to give 4.1 g (85%) **3**.

Method B. A mixture of 0.42 g **8** (2.62 mmol) and 0.42 g potassium ethylxanthogenate (2.62 mmol) in 10 cm^3 2-propanol was refluxed for 4 h. After cooling, the precipitate was filtered off, washed with 2-propanol and acetone, and dried to give 0.45 g (71%) **3**.

In both cases the salts were analytically pure. If necessary, additional purification could be achieved by recrystallization from *Et*OH–H_2O. Mp 356–358°C; ^{1}H NMR (D_2O): $\delta = 8.32$ (s, H-5), 7.29 (m, H-7, H-8, H-9, H-10) ppm.

2-Thio[1,2,4]triazolo[1,5-c]quinazoline (**4**, $C_9H_6N_4S$)
To a solution of 1.2 g **3** (5 mmol) in 30 cm^3 H_2O was added 10% aqueous HCl to a $pH = 3$–4. The resulting precipitate was filtered off, thoroughly washed with H_2O, and recrystallized from toluene to give 1.0 g (99%) **4**. Mp 262–264°C; ^{1}H NMR: $\delta = 9.50$ (s, H-5), 8.42 (d, $J = 7.6$ Hz, H-10), 8.07 (d, $J = 7.7$ Hz, H-7), 7.97 (t, $J = 7.6$ Hz, H-8), 7.84 (t, $J = 7.6$ Hz, H-9) ppm; MS (APCI): $m/z = 205$, 203 (MH^+); MS (EI): $m/z = 204$ (11.6), 203 (33.9), 202 ($M^{+\bullet}$, 100.0), 176 (4.8), 175 (25.6), 173 (4.6), 160 (7.5), 157 (3.0), 156 (30.4), 146 (9.0), 144 (17.0), 143 (4.9), 130 (11.2), 129 (73.8), 120 (3.7), 117 (14.0), 116 (8.2), 115 (6.5), 114 (15.6), 103 (15.7), 102 (67.4), 101 (4.2), 90 (12.8), 89 (8.7), 88 (26.7), 87 (15.9), 86 (4.4), 85 (3.3), 77 (3.6), 76 (24.6), 75 (31.3), 74 (21.7), 73 (12.4), 70 (4.2), 65 (3.4), 64 (9.2), 63 (11.6), 62 (15.4), 61 (4.5), 59 (3.1), 58 (8.1), 52 (5.6), 51 (14.7), 50 (11.8), 46 (3.2), 45 (21.6), 44 (16.8), 42 (5.8), 39 (8.7), 38 (6.1), 37 (3.3), 32 (8.2), 28 (21.0).

General Procedure for the Synthesis of 2-(Alkylthio)-[1,2,4]triazolo[1,5-c]quinazolines ***5a–5d***
To a suspension of 2.4 g **3** (10 mmol) in 15 cm^3 *Et*OH were added 11 mmol of an appropriate alkyl halide (ethyl iodide, propyl iodide, benzyl chloride), or phenacyl chloride, and the resulting mixture was heated for 30 min. On cooling, five-fold excess of H_2O was added. The crystalline precipitate formed was filtered off and recrystallized from an appropriate solvent to yield the target **5a**–**5d**.

2-(Ethylthio)[1,2,4]triazolo[1,5-c]quinazoline (**5a**, $C_{11}H_{10}N_4S$)
Yield 66% (1.5 g, from 2-propanol); mp 120–122°C; ^{1}H NMR: $\delta = 9.53$ (s, H-5), 8.39 (d, $J = 7.7$ Hz, H-10), 8.04 (d, $J = 7.7$ Hz, H-7), 7.95 (t, $J = 7.6$ Hz, H-8), 7.82 (t, $J = 7.6$ Hz, H-9), 3.31 (q, $J = 7.3$ Hz, SCH_2), 1.44 (t, $J = 7.3$ Hz, CH_3) ppm; MS (APCI): $m/z = 232$, 231 (MH^+).

2-(Propylthio)[1,2,4]triazolo[1,5-c]quinazoline (**5b**, $C_{12}H_{12}N_4S$)
Yield 58% (1.4 g, from 2-propanol); mp 98–100°C; ^{1}H NMR: $\delta = 9.50$ (s, H-5), 8.37 (d, $J = 7.7$ Hz, H-10), 8.02 (d, $J = 7.7$ Hz, H-7), 7.92 (t, $J = 7.6$ Hz, H-8), 7.79 (t, $J = 7.6$ Hz, H-9), 3.25 (t, $J = 7.3$ Hz, SCH_2), 1.79 (sxt, $J = 7.3$ Hz, CH_2CH_3), 1.02 (t, $J = 7.3$ Hz, CH_3) ppm; ^{13}C NMR: $\delta = 165.92$ (5-C), 151.17 (2-C), 142.87 (10b-C), 138.57 (6a-C), 132.86 (8-C), 129.45 (7-C), 128.86 (9-C), 123.73 (10-C), 117.17 (10a-C), 33.32 (SCH_2), 22.93 (CH_2CH_3), 13.60 (CH_3) ppm; MS (APCI): $m/z = 246$, 245 (MH^+); MS (EI): $m/z = 246$ (3.6), 245 (25.3), 244 ($M^{+\bullet}$, 29.5) 229 (18.5), 217 (4.3), 216 (30.7), 215 (36.9), 212 (10.5), 211 (71.6), 205 (3.3), 204 (34.7), 203 (70.6), 202 (100.0), 201 (11.5), 198 (16.5), 197 (76.8), 195 (8.5), 189 (9.7), 188 (10.5), 187 (20.8), 186 (3.3), 185 (13.9), 184 (66.1), 183 (5.7), 176 (9.3), 175 (40.1), 174 (3.2), 173 (15.8), 171 (34.7), 170 (9.5), 169 (5.0), 160 (3.2), 157 (3.9), 156 (17.4), 146 (7.7), 144 (5.9), 130 (7.2), 129 (41.7), 103 (3.5), 102 (17.7), 88 (4.3), 76 (9.3), 75 (12.2), 74 (9.5), 64 (7.0), 63 (8.6), 62 (9.2), 59 (7.8), 58 (4.9), 51 (4.7).

2-(Benzylthio)[1,2,4]triazolo[1,5-c]quinazoline (**5c**, $C_{16}H_{12}N_4S$)
Yield 71% (2.1 g, from 2-propanol); mp 129–131°C; ^{1}H NMR: $\delta = 9.53$ (s, H-5), 8.40 (d, $J = 7.7$ Hz, H-10), 8.04 (d, $J = 7.7$ Hz, H-7), 7.93 (t, $J = 7.6$ Hz, H-8), 7.81 (t, $J = 7.6$ Hz, H-9), 7.51 (d, $J = 7.4$ Hz, 2H, Ph), 7.30 (m, 3H, Ph), 4.57 (s, SCH_2) ppm; MS (APCI): $m/z = 293$ (MH^+).

1-Phenyl-2-([1,2,4]triazolo[1,5-c]quinazolin-2-ylthio)-1-ethanone (**5d**, $C_{17}H_{12}N_4OS$)
Yield 76% (2.4 g, from dioxane–H_2O); mp 169–171°C; ^{1}H NMR: $\delta = 9.50$ (s, H-5), 8.33 (d, $J = 7.7$ Hz, H-10), 8.10 (d, $J = 7.6$ Hz, 2H, Ph), 8.04 (d, $J = 7.7$ Hz, H-7), 7.93 (t, $J = 7.6$ Hz, H-8), 7.80 (t, $J = 7.6$ Hz, H-9), 7.71 (t, 1H, $J = 7.6$ Hz, Ph), 7.60 (t, $J = 7.6$ Hz, 2H, Ph), 5.09 (s, SCH_2) ppm; ^{13}C NMR: $\delta = 165.22$ (5-C), 151.20 (2-C), 142.85 (10b-C), 138.53 (6a-C), 136.03 (1-Ph), 134.22 (4-Ph), 132.96 (8-C), 129.52 (7-C), 129.37 (2,6-Ph), 128.88 (9-C, 3,5-Ph), 123.69 (10-C), 117.09 (10a-C), 40.09 (CH_2) ppm; MS (APCI): $m/z = 323$, 321 (MH^+); MS (EI): $m/z = 322$ (8.7), 321 (42.3), 320 ($M^{+\bullet}$, 52.8), 319 (28.8), 302 (7.2), 292 (15.8), 287 (4.0), 218 (5.1), 217 (8.7), 216 (19.5), 215 (100.0), 203 (4.7), 202 (8.3), 201 (8.3), 188 (5.8), 187 (13.1), 129 (5.2), 105 (26.8), 77 (20.1).

2-([1,2,4]Triazolo[1,5-c]quinazolin-2-ylthio)acetic acid (**5e**, $C_{11}H_8N_4O_2S$)
To a solution of 2.4 g **3** (10 mmol) in 15 cm^3 H_2O was added a solution of 1.04 g chloroacetic acid (11 mmol) in 12 cm^3 5% aqueous KOH and the resulting mixture was heated for 30 min. On cooling, all insoluble materials were filtered off from the reaction mixture. To the resulting solution was added 10% aqueous HCl to a $pH = 3$–4, and the precipitate was filtered off and washed well with H_2O. The obtained substance was analytically pure. Yield 75% (1.9 g); mp 232–234°C; ^{1}H NMR: $\delta = 11.43$ (br.s, COOH), 9.30 (s, H-5), 8.40 (d, $J = 7.7$ Hz, H-10), 7.98 (d, $J = 7.7$ Hz, H-7), 7.84 (t, $J = 7.6$ Hz, H-8), 7.73 (t, $J = 7.6$ Hz, H-9), 4.05 (s, CH_2) ppm; ^{13}C NMR: $\delta = 170.09$ (COOH), 165.17 (5-C), 151.20 (2-C), 142.79 (10b-C), 138.49 (6a-C), 132.90 (8-C), 129.47 (7-C), 128.83 (9-C), 123.67 (10-C), 117.04 (10a-C), 34.02 (CH_2) ppm; MS (APCI): $m/z = 263$, 261 (MH^+); MS (EI): m/z (%) = 261 (8), 260 ($M^{+\bullet}$, 12), 218 (15), 217 (35), 216 (100), 215 (77), 189 (7), 188 (4), 187 (4), 184 (10), 183 (4), 171 (17), 170 (3), 130 (5), 129 (17), 102 (10), 75 (5).

4-Imino-3(4H)-quinazolinamine (**8**, $C_8H_8N_4$)
A mixture of 1.18 g **6** (10 mmol) in 5 cm^3 triethyl orthoformate was refluxed for 3 h, and then the excess reagent was distilled off *in vacuo*. The solid thus obtained was treated with 1 cm^3 of refluxing 99% hydrazine hydrate in 10 cm^3 *Et*OH for 1 h. The reaction mixture was allowed to cool. The solid obtained was filtered off, washed with *Et*OH, and recrystallized from toluene–methanol to give 0.9 g (56%) **8**. Mp 200–202°C; ^{1}H NMR: $\delta = 9.55$ (s, NH), 8.06 (s, H-2), 7.37–7.62 (m, H-5, H-6, H-7, H-8), 4.76 (s, NH_2) ppm; MS (APCI): $m/z = 161$ (MH^+).

X-Ray Crystal Structure Determination of ***5b***
Single crystals were obtained by slow evaporation of ethanol solution. The crystals of $C_{12}H_{12}N_4S$ are triclinic. At 100 K $a = 7.944(1)$, $b = 8.239(1)$, $c = 10.314(2)$ Å, $\alpha = 95.86(2)$, $\beta = 111.32(1)$, $\gamma = 109.65(1)°$, $V = 572.5(2)$ Å^3, $M_r = 244.32$, $Z = 2$, space group $P\bar{1}$, $d_{calc} = 1.417$ g/cm^3, $\mu(MoK_\alpha) = 0.264$ mm^{-1}, $F(000) = 256$. Intensity of 6786 reflections (1892 independent, $R_{int} = 0.027$) 0.027) were measured on an automatic "X-calibur" diffractometer (graphite monochromated MoK_α radiation, ω scaning, $2\Theta_{max} = 50°$). The structure was solved by direct method using SHELXTL package [17]. Positions of hydrogen atoms were located from electron density difference maps and refined isotropically. Full-matrix least-squares refinement against F^2 in anisotropic approximation using 1828 reflections was converged to $R_1 = 0.028$ (for 1789 reflections with $F > 4\sigma(F)$), $wR_2 = 0.077$, $S = 1.067$. Atomic coordinates and crystallographic parameters have been deposited at the Cambridge Crystallographic Data Centre (CCDC 606659). These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

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