Synthesis and molecular structure of 5-(1-aminoisoindolenin-3-ylidene-amino)-1,3,4-thiadiazole-2(3*H*)-thione

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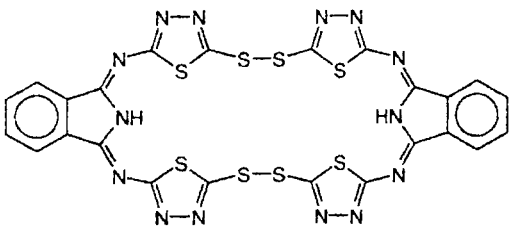
The structure of 5-(1-aminoisoindolenin-3-ylideneamino)-1,3,4-thiadiazole-2(3*H*)-thione was studied by X-ray diffraction analysis and UV and IR spectroscopy. In the crystal, the molecule of the title compound is planar in spite of the substantially shortened intramolecular N...S contact. The factors that determine the structure of the heterocycle, namely, the nature of the N...S interaction, conjugation effects, and the tautomerism, were analyzed based on quantum-chemical calculations performed by the AM1 method.

Key words: 1,3,4-thiadiazole-2(3*H*)-thione, isoindolenine, crystal solvate, molecular structure, X-ray diffraction analysis.

Compounds whose molecules consist of two or several heterocycles of different nature are convenient models for studying the mutual effects of heterocycles both on tautomeric conversions and on the properties of these compounds.

The aim of this work was to study compounds containing thiadiazole and indole fragments. It is known that thiadiazoles are objects of studies devoted to the search for efficient antitumor drugs[1] and are used as diuretics and sulfanilamide drugs.[2] Isoindole and its derivatives are also of obvious practical interest. It will suffice to mention that phthalocyanines, which have long been used as dyes,[3] contain isoindole fragments. A number of derivatives of isoindole were recommended as drugs.[4] We chose macroheterocycle **1**[5] as the first object of studies.



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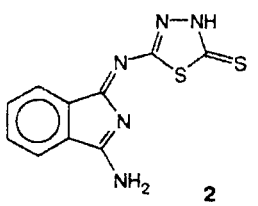

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However, we failed to obtain single crystals of this compound because of its thermal lability in solution. When we attempted to crystallize compound **1** from an aqueous DMF solution, it underwent hydrolysis to form 5-(1-aminoisoindolenin-3-ylideneamino)-1,3,4-thiadiazole-2(3*H*)-thione **2**. However, compound **2** models the major structural features of the fragments of **1**. Therefore, we studied the molecular structure of **2**.

Results and Discussion

It was found that when compound **1** was recrystallized from aqueous DMF, a new compound was formed, which differs from the initial compound in physicochemical and spectral properties. We succeeded in preparing a single crystal of this compound. It appeared that we obtained 5-(1-aminoisoindolenin-3-ylideneamino)-1,3,4-thiadiazole-2(3*H*)-thione (**2**), which is thermodynamically stable under these conditions. Compound **2** was, apparently, formed as a result of hydrolysis of **1**.

Compound **2** was obtained as a yellow powder insoluble in heptane, benzene, water, and dilute acids, soluble in DMF, pyridine, and aqueous solutions of alkalis, and poorly soluble in alcohols. The UV spectrum of compound **2** in DMF has an intense absorption band with maxima at 420 and 437 nm. The splitting of this

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band may be caused by the isoindole-isoindolenine tautomerism of thione **2**. In going from DMF to a 0.1 *M* NaOH solution, the absorption band shifts substantially to the short-wavelength region with a simultaneous increase in its intensity. In this case, the spectrum is characterized by the presence of only one maximum at 371 nm. Apparently, thione **2** reacts with NaOH at the S atom of the thioketone group to form the sodium salt, which occurs only in the thiadiazole form. This form is characterized by an absorption band shifted even more substantially to the short-wavelength region. The position and intensity of the absorption band in the UV spectrum allow one to assign it to the π—π^* type and provide evidence that a general conjugated system occurs in compound **2**.

The structure of thione **2** was studied by X-ray diffraction analysis (Fig. 1, Tables 1—3).

In the crystal, compound **2** occurs as a 1 : 1 : 1 solvate with DMF and water (Fig. 2). There are two structural units of this composition per asymmetric unit. Molecules **2** in these solvates (*A* and *B*) have close geometric parameters. It should be noted that the isoindolenine and thiadiazoline fragments are coplanar within 0.01 Å in spite of the shortened intramolecular S(1)...N(1) contact, 2.74 Å (the sum of the van der Waals radii is 3.34 Å).[6]

The lengths of the C(10)—S(2) [1.671(5) Å (*A*, *B*)] C(10)—N(5) [1.326(6) Å (*A*) and 1.333(5) Å (*B*)], and

Table 1. Coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$) of nonhydrogen atoms in the structure of **2**

Atom	*x*	*y*	*z*	U_{eq}/Å^2
S(1*A*)	13550(2)	10841(1)	5389(1)	49(1)
S(2*A*)	13059(2)	9398(2)	6482(1)	62(1)
N(1*A*)	14575(5)	12569(4)	4511(2)	47(1)
N(2*A*)	16791(6)	15023(4)	4434(2)	60(1)
N(3*A*)	11989(5)	10150(4)	4257(2)	48(1)
N(4*A*)	10982(5)	8368(4)	4896(2)	52(1)
N(5*A*)	11252(5)	8185(4)	5451(2)	49(1)
C(1*A*)	11882(7)	11449(6)	3093(2)	53(1)
C(2*A*)	12162(7)	12321(6)	2638(2)	61(1)
C(3*A*)	13581(7)	13773(6)	2686(2)	59(1)
C(4*A*)	14754(7)	14396(6)	3184(2)	51(1)
C(5*A*)	14471(6)	13526()45	3635(2)	43(1)
C(6*A*)	15367(6)	13768(5)	4223(2)	44(1)
C(7*A*)	13161(6)	11494(5)	4148(2)	43(1)
C(8*A*)	13049(6)	12066(5)	3590(2)	45(1)
C(9*A*)	12100(6)	9726(5)	4795(2)	44(1)
C(10*A*)	12518(6)	9341(5)	5791(2)	45(1)
S(1*B*)	7606(2)	13645(1)	680(1)	46(1)
S(2*B*)	10630(2)	14832(1)	1696(1)	59(1)
N(1*B*)	4490(5)	12173(4)	−126(1)	42(1)
N(2*B*)	1939(5)	9819(4)	−221(2)	54(1)
N(3*B*)	6575(5)	14646(4)	−347(1)	41(1)
N(4*B*)	9100(5)	16262(4)	279(1)	41(1)
N(5*B*)	10090(5)	16280(4)	785(1)	42(1)
C(1*B*)	3927(7)	13799(6)	−1474(2)	54(1)
C(2*B*)	2493(7)	13103(6)	−1922(2)	62(1)
C(3*B*)	1103(7)	11677(6)	−1896(2)	63(1)
C(4*B*)	1069(7)	10872(5)	−1420(2)	52(1)
C(5*B*)	2480(6)	11555(5)	−981(2)	43(1)
C(6*B*)	2940(6)	11108(5)	−418(2)	42(1)
C(7*B*)	5112(6)	13356(5)	−466(2)	39(1)
C(8*B*)	3887(6)	12993(5)	−1010(2)	43(1)
C(9*B*)	7723(6)	14927(4)	157(2)	37(1)
C(10*B*)	9576(6)	15044(5)	1075(2)	43(1)
N(1*AS*)	14785(5)	20350(4)	1740(2)	55(1)
O(1*AS*)	12947(5)	19134(4)	927(1)	60(1)
C(1*AS*)	14108(7)	20271(6)	1213(2)	56(1)
C(2*AS*)	16246(10)	21739(7)	2034(3)	87(2)
C(3*AS*)	14177(10)	19013(6)	2063(3)	79(2)
N(1*BS*)	8611(5)	4188(4)	6393(2)	50(1)
O(1*BS*)	8615(5)	5351(4)	5587(1)	64(1)
C(1*BS*)	7992(7)	4264(6)	5873(2)	58(1)
C(2*BS*)	7716(9)	2853(6)	6698(2)	73(2)
C(3*BS*)	10228(8)	5444(6)	6697(2)	70(2)
O(1*AW*)	1578(5)	2346(4)	6362(2)	74(1)
O(2*AW*)	−1696(6)	7607(4)	−920(2)	73(1)

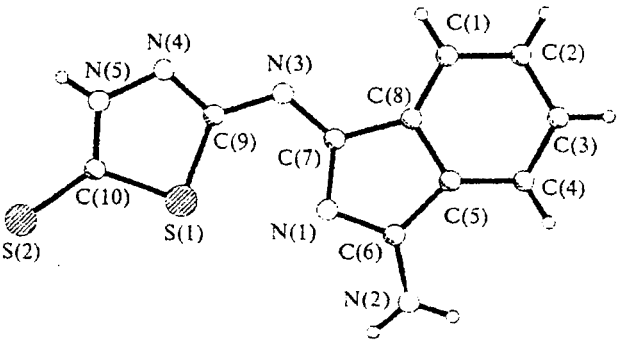


Fig. 1. Structure of compound **2**.

Table 2. Bond lengths (*d*) in the structure of **2**

Bond	*d*/Å	Bond	*d*/Å
S(1*A*)—C(10*A*)	1.736(4)	S(1*B*)—C(10*B*)	1.741(4)
S(1*A*)—C(9*A*)	1.749(5)	S(1*B*)—C(9*B*)	1.760(4)
S(2*A*)—C(10*A*)	1.670(5)	S(2*B*)—C(10*B*)	1.672(5)
N(1*A*)—C(6*A*)	1.331(5)	N(1*B*)—C(6*B*)	1.338(5)
N(1*A*)—C(7*A*)	1.367(5)	N(1*B*)—C(7*B*)	1.379(5)
N(2*A*)—C(6*A*)	1.311(5)	N(2*B*)—C(6*B*)	1.313(5)
N(3*A*)—C(7*A*)	1.309(5)	N(3*B*)—C(7*B*)	1.307(5)
N(3*A*)—C(9*A*)	1.377(6)	N(3*B*)—C(9*B*)	1.370(5)
N(4*A*)—C(9*A*)	1.298(5)	N(4*B*)—C(9*B*)	1.303(5)
N(4*A*)—N(5*A*)	1.362(5)	N(4*B*)—N(5*B*)	1.356(5)
N(5*A*)—C(10*A*)	1.329(5)	N(5*B*)—C(10*B*)	1.331(5)
C(1*A*)—C(8*A*)	1.377(6)	C(1*B*)—C(8*B*)	1.371(6)
C(1*A*)—C(2*A*)	1.385(6)	C(1*B*)—C(2*B*)	1.395(7)
C(2*A*)—C(3*A*)	1.386(7)	C(2*B*)—C(3*B*)	1.380(7)
C(3*A*)—C(4*A*)	1.381(7)	C(3*B*)—C(4*B*)	1.395(7)
C(4*A*)—C(5*A*)	1.376(6)	C(4*B*)—C(5*B*)	1.371(6)
C(5*A*)—C(8*A*)	1.392(6)	C(5*B*)—C(8*B*)	1.395(6)
C(5*A*)—C(6*A*)	1.474(6)	C(5*B*)—C(6*B*)	1.479(6)
C(7*A*)—C(8*A*)	1.473(6)	C(7*B*)—C(8*B*)	1.472(6)
N(1*AS*)—C(1*AS*)	1.307(6)	N(1*BS*)—C(1*BS*)	1.304(6)
N(1*AS*)—C(2*AS*)	1.458(6)	N(1*BS*)—C(3*BS*)	1.454(6)
N(1*AS*)—C(3*AS*)	1.455(6)	N(1*BS*)—C(2*BS*)	1.460(6)
O(1*AS*)—C(1*AS*)	1.229(5)	O(1*BS*)—C(1*BS*)	1.230(5)

Table 3. Bond angles (ω) in the structure of **2**

Angle	ω/deg	Angle	ω/deg
C(10*A*)—S(1*A*)—C(9*A*)	89.5(2)	C(10*B*)—S(1*B*)—C(9*B*)	89.5(2)
C(6*A*)—N(1*A*)—C(7*A*)	106.7(4)	C(6*B*)—N(1*B*)—C(7*B*)	106.2(4)
C(7*A*)—N(3*A*)—C(9*A*)	119.1(4)	C(7*B*)—N(3*B*)—C(9*B*)	120.7(4)
C(9*A*)—N(4*A*)—N(5*A*)	110.1(4)	C(9*B*)—N(4*B*)—N(5*B*)	109.8(3)
C(10*A*)—N(5*A*)—N(4*A*)	119.0(4)	C(10*B*)—N(5*B*)—N(4*B*)	120.0(3)
C(8*A*)—C(1*A*)—C(2*A*)	118.1(5)	C(8*B*)—C(1*B*)—C(2*B*)	116.6(5)
C(1*A*)—C(2*A*)—C(3*A*)	120.6(5)	C(1*B*)—C(2*B*)—C(3*B*)	121.6(5)
C(4*A*)—C(3*A*)—C(2*A*)	121.5(5)	C(4*B*)—C(3*B*)—C(2*B*)	121.3(5)
C(5*A*)—C(4*A*)—C(3*A*)	117.8(5)	C(5*B*)—C(4*B*)—C(3*B*)	117.1(4)
C(4*A*)—C(5*A*)—C(8*A*)	121.0(4)	C(4*B*)—C(5*B*)—C(8*B*)	121.3(4)
C(4*A*)—C(5*A*)—C(6*A*)	134.5(4)	C(4*B*)—C(5*B*)—C(6*B*)	134.0(4)
C(8*A*)—C(5*A*)—C(6*A*)	104.5(4)	C(8*B*)—C(5*B*)—C(6*B*)	104.7(4)
N(2*A*)—C(6*A*)—N(1*A*)	124.1(4)	N(2*B*)—C(6*B*)—N(1*B*)	123.4(4)
N(2*A*)—C(6*A*)—C(5*A*)	123.5(4)	N(2*B*)—C(6*B*)—C(5*B*)	124.1(4)
N(1*A*)—C(6*A*)—C(5*A*)	112.4(4)	N(1*B*)—C(6*B*)—C(5*B*)	112.5(4)
N(3*A*)—C(7*A*)—C(1*A*)	127.5(4)	N(3*B*)—C(7*B*)—C(1*B*)	127.1(4)
N(3*A*)—C(7*A*)—C(8*A*)	122.0(4)	N(3*B*)—C(7*B*)—C(8*B*)	122.1(4)
N(1*A*)—C(7*A*)—C(8*A*)	110.4(4)	N(1*B*)—C(7*B*)—C(8*B*)	110.8(4)
C(1*A*)—C(8*A*)—C(5*A*)	121.1(4)	C(1*B*)—C(8*B*)—C(5*B*)	122.0(4)
C(1*A*)—C(8*A*)—C(7*A*)	133.0(4)	C(1*B*)—C(8*B*)—C(7*B*)	132.1(4)
C(5*A*)—C(8*A*)—C(7*A*)	105.9(4)	C(5*B*)—C(8*B*)—C(7*B*)	105.9(4)
N(4*A*)—C(9*A*)—N(3*A*)	118.8(4)	N(4*B*)—C(9*B*)—N(3*B*)	119.4(4)
N(4*A*)—C(9*A*)—S(1*A*)	113.6(3)	N(4*B*)—C(9*B*)—S(1*B*)	113.4(3)
N(3*A*)—C(9*A*)—S(1*A*)	127.6(3)	N(3*B*)—C(9*B*)—S(1*B*)	127.2(3)
N(5*A*)—C(10*A*)—S(2*A*)	127.6(3)	N(5*B*)—C(10*B*)—S(2*B*)	127.1(3)
N(5*A*)—C(10*A*)—S(1*A*)	107.7(3)	N(5*B*)—C(10*B*)—S(1*B*)	107.2(3)
S(2*A*)—C(10*A*)—S(1*A*)	124.7(3)	S(2*B*)—C(10*B*)—S(1*B*)	125.7(3)
C(1*AS*)—N(1*AS*)—C(2*AS*)	122.8(4)	C(1*BS*)—N(1*BS*)—C(3*BS*)	121.1(4)
C(1*AS*)—N(1*AS*)—C(3*AS*)	121.3(4)	C(1*BS*)—N(1*BS*)—C(2*BS*)	121.9(4)
C(2*AS*)—N(1*AS*)—C(3*AS*)	115.9(5)	C(3*BS*)—N(1*BS*)—C(2*BS*)	117.0(4)
O(1*AS*)—C(1*AS*)—N(1*AS*)	127.1(5)	O(1*BS*)—C(*BS*)—N(1*BS*)	126.7(4)

C(10)—S(1) [1.737 Å (*A*) and 1.746 Å (*B*)] bonds are close to the corresponding values in derivatives of thiadiazoline,[7–9] which confirms that the C(10)=S(2) bond is double. The absence of hydrogen atoms at the N(1) and N(3) atoms and the presence of two hydrogen atoms at the N(2) atom suggest that the N(3)=C(7) and N(1)=C(6) bonds are double and the N(2)—C(6) bond is single. However, a comparison of the bond lengths in the N(3)=C(7)—N(1)=C(6)—N(2) fragment of molecule **2** and in analogous derivatives of isoindole[10–12] suggests a substantial contribution of the N(3)=C(7)—N(1)—C(6)=N(2) structure. This structure with the exocyclic double C=N bonds can occur in one of the two tautomers of **2** (**2a** and **2b**). This suggestion was confirmed by the results of quantum-chemical calculations, which were performed by the semiempirical AM1 method,[13] of the structures of the two tautomers of the compound. The N(2)—C(6) and N(1)—C(6) bond lengths in **2a** have close values (Table 4), which is indicative of the substantial contribution of the bipolar resonance structure $^{+}NH_2$=C—N^{-}—C=N to the structure of the fragment under consideration. According to calculations, in the case of tautomer **2b**, the structure with the exocyclic

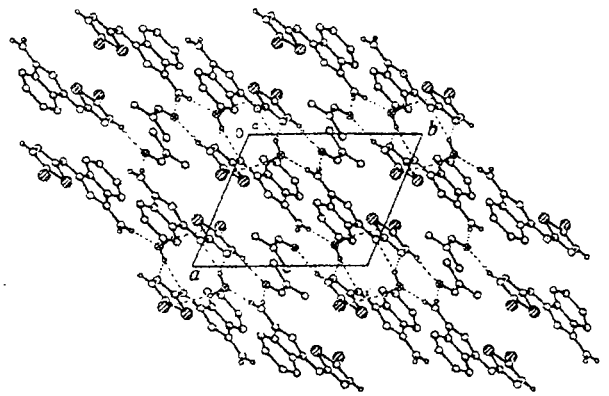


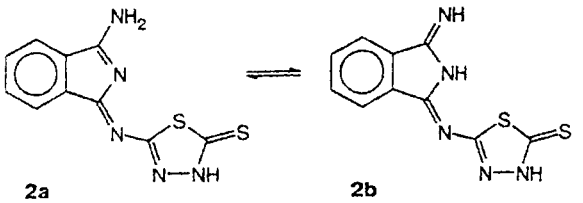
Fig. 2. Crystal packing of solvate of **2**.

Table 4. Calculated bond lengths (*d*) in the N(2)C(6)N(1)C(7)N(3)C(9)S(1) fragment of tautomers **2a** and **2b**

Bond	*d*/Å		Bond	*d*/Å	
	2a	**2b**		**2a**	**2b**
N(2)—C(6)	1.359	1.285	C(7)—N(3)	1.302	1.311
C(6)—N(1)	1.349	1.440	N(3)—C(9)	1.397	1.384
N(1)—C(7)	1.443	1.408	C(9)—S(1)	1.769	1.758

double C=N bonds makes the major contribution. The experimental values of the bond lengths in the fragment under consideration have intermediate values compared to the calculated data on two tautomers (see Table 4).

Therefore, it can be suggested that both tautomers (**2a** and **2b**) occur in the crystal, and the geometry of molecule **2** determined by X-ray diffraction study is the geometry averaged over two tautomers. Unfortunately, we failed to reveal the position of the H atom at the N(1) atom, which corresponds to tautomer **2b**.



2a **2b**

In the crystal of compound **2**, DMF and water molecules form a three-dimensional framework (see Fig. 2) through the hydrogen bonds H(2*B*N)...O(1*BS*)′ ($1 + x$, $1 + y$, z) (O...H, 2.06 Å; O...H—N, 175°), H(2*A*N(2))...O(1*AS*)′ ($-1 + x$, $-1 + y$, z) (O...H, 2.09 Å; O...N—H, 157°), H(5*A*N)...O(1*BS*) (O...H, 1.93 Å; O...H—N, 158°), H(5*B*N)...O(1*AS*) (O...H, 1.93 Å; O...H—N, 157°), and H(1*AW*(2))...N(3)′ ($1 - x$, $1 - y$, $1 - z$) (N...H, 2.13 Å; O...H—N, 163°) and the shortened intermolecular contacts H(2N)...O(1*AW*), ($2 - x$, $2 - y$, $1 - z$) (2.30 Å; the sum of the van der Waals radii[8] is 2.45 Å), H(1*AW*(1))...S(2)′ ($-1 - x$, $-1 - y$, z) (2.51 Å; 3.00 Å)), and H(2*AW*(2))...S(2*B*) (2.39 Å).

The shortened intramolecular contact between the S(1) and N(1) atoms is of particular interest. The distance between these atoms is substantially shorter (by 0.6 Å) than the sum of the van der Waals radii, which suggests the presence of specific interactions between them. Quantum-chemical calculations of tautomeric forms **2a** and **2b** revealed the minimum on the potential energy surface of the molecule, which corresponds to the planar structure with the shortened S...N contact. Therefore, the observed flattening of molecule **2** in the crystal is caused by intramolecular factors rather than by intermolecular interactions. Among these factors may be the interaction of the lone electron pair of the N atom with the antibonding orbital of the C—S bond in structure **2a**, the intramolecular N—H...S hydrogen bond in structure **2b**, and the electrostatic interaction of the opposite charged N(1) and S(1) atoms.

Analysis of the orbital structure of compound **2a** demonstrated that there are no interactions between AOs of the S and N atoms. This is also supported by the zero values of the Mulliken population coefficients of the overlap between the AOs of these atoms and by the zero value of the order of the N(1)...S(1) bond. Therefore, there are no $n \rightarrow \sigma^*$ interactions in structure **2a**.

The calculated values of the charges on the N(1) and S(1) atoms have opposite signs in both tautomeric forms under consideration: N(1), −0.29 (**2a**) and −0.38 (**2b**); and S(1), 0.43 (**2a**) and 0.22 (**2b**). The presence of the positive charge on the S atom excludes the possibility of formation of an intramolecular N—H...S hydrogen bond in structure **2b**. However, the large opposite charges on the atoms under consideration result in a strong electrostatic attraction, which, apparently, stabilizes the planar conformation of molecule **2** with the shortened N...S contact in both tautomeric forms.

Experimental

Synthesis and purification of macroheterocyclic compound **1**, which is the starting compound for preparing thione **2**, was carried out using a known procedure.[14]

5-(1-Aminoisoindolenin-3-ylideneamino)-1,3,4-thiadiazole-2(3*H*)-thione (2) was prepared by boiling compound **1** in aqueous DMF for 1 h at the **2** : DMF ratio of 1 : 20 (wt.%). The solution was cooled and then kept at 20 °C for a long period. Thione **2** was precipitated as large platelet-like crystals suitable for X-ray diffraction study. Crystals of **2**, which were powdered and kept for 2 h at the residual pressure of 3 kPa and 150 °C, were used for spectral and physicochemical studies.

The UV spectra were recorded on a Specord M-40 spectrophotometer. The IR spectra were recorded on a Specord M-80 spectrometer.

Compound **2** was obtained as a yellow powder, m.p. 251—253 °C (with decomp.). UV (DMF), λ_{max}/nm (log ε): 420.0 (4.32), 437.0; (0.1 *N* NaOH) 371.0 (4.55). IR (KBr pellets), ν/cm^{-1}: 3140 (N—H), 3080 (C—H), 1636 (C=C), 1620 (C=N increased), 1228 (C=S), 1183 (C—S), 780 (N—H), 762 (out-of-plane C—H vibration), 468 (C—S in the heterocycle). Found (%): C, 45.42; H, 2.81; N, 26.21; S, 24.33. $C_{10}H_7N_5S_2$. Calculated (%): C, 45.96; H, 2.70; N, 26.50; S, 24.54.

Crystals of **2** ($2C_{10}H_7N_5S_2 \cdot 2C_3H_7NO \cdot 2H_2O$) are triclinic. At 20 °C, a = 7.823(3) Å, b = 9.389(4) Å, c = 24.314(9) Å, α = 91.32(3)°, β = 97.92(3)°, γ = 112.13(3)°, V = 1633(1) Å^3, d_{calc} = 1.438 g cm^{-3}, space group $P\bar{1}$, Z = 2. The unit cell parameters and intensities of 6913 independent reflections (R_{int} = 0.072) were measured on an automated Siemens P3/PC diffractometer (λ(Mo-Kα) radiation, graphite monochromator, θ/2θ scanning technique, $2\theta_{max}$ = 54°).

The structure was solved by the direct method with the use of the SHELXTL PLUS program package.[15] The positions of hydrogen atoms were located from the difference electron density synthesis and refined using the riding model (U_{iso} were not fixed). The refinement based on F^2 was carried out by the full-matrix least-squares method with anisotropic thermal parameters for nonhydrogen atoms using 4063 reflections and converged to wR_2 = 0.141 (R_1 = 0.061 using 2971 reflections with $F > 4\sigma(F)$, S = 1.06). The coordinates of nonhydrogen atoms are given in Table 1. The bond lengths and bond angles are listed in Tables 2 and 3, respectively.

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