

## 4-HYDROXYQUINOLONES-2. 247\*. 4-HYDROXY-2-OXO-1,2-DIHYDROQUINOLINE OR 4-HYDROXY-2,2-DIOXO-1H-2λ<sup>6</sup>,1-BENZOTHAZINE?

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*Methyl 2-[(2-{[2-(methoxycarbonyl)phenyl]amino}-2-oxoethyl)sulfonyl](methyl)amino}benzoate cyclized in the presence of bases giving exclusively 2-substituted anilides of 4-hydroxy-1-methyl-2-oxo-1,2-dihydroquinoline-3-sulfonic acid, regardless of the strength of the base used.*

**Keywords:** 4-hydroxy-2,2-dioxo-1H-2λ<sup>6</sup>,1-benzothiazines, 4-hydroxy-2-oxo-1,2-dihydroquinolines, heterocyclization.

During the base-catalyzed heterocyclization of alkyl 2-[2-(alkoxycarbonyl)phenylsulfamoyl]acetates into the esters of 1-R-4-hydroxy-2,2-dioxo-1H-2λ<sup>6</sup>,1-benzothiazine-3-carboxylic acids [2] it was observed that formation of 2,1-benzothiazine ring was somewhat more difficult than formation of quinolines from alkyl 2-[2-(alkoxycarbonyl)phenylcarbamoyl]acetates.

In the current investigation, we attempted to confirm this observation with objective experimental data. For this purpose, we performed a simple and elegant experiment, allowing to estimate quantitatively the differences of how easily the structurally related 4-hydroxy-2-oxo-1,2-dihydroquinoline and 4-hydroxy-2,2-di-oxo-1H-2λ<sup>6</sup>,1-benzothiazine heterocycles were formed. The starting material for this study was the dianilide of sulfoacetic acid **2** obtained by the reaction of methyl *N*-methylantranylate (**1**) with (chlorosulfonyl)acetyl chloride. The acylation in these experiments was performed under conditions designed to exclude even partial intramolecular cyclization by avoiding sufficiently strong bases (such as tertiary alkylamines) commonly used in reactions of this type for neutralization of HCl evolved. Dianilide **2** can theoretically convert by the action of basic catalysts into either benzothiazine **3** or quinoline **4**, or a mixture of these compounds, and the unique structure of compound **2** creates identical conditions for formation of both heterocycles, that would be difficult to achieve otherwise.

Further, the experiment involves essentially a qualitative and quantitative analysis of the reaction mixtures formed by treatment of dianilide **2** with bases. This study relies of unequivocal structural

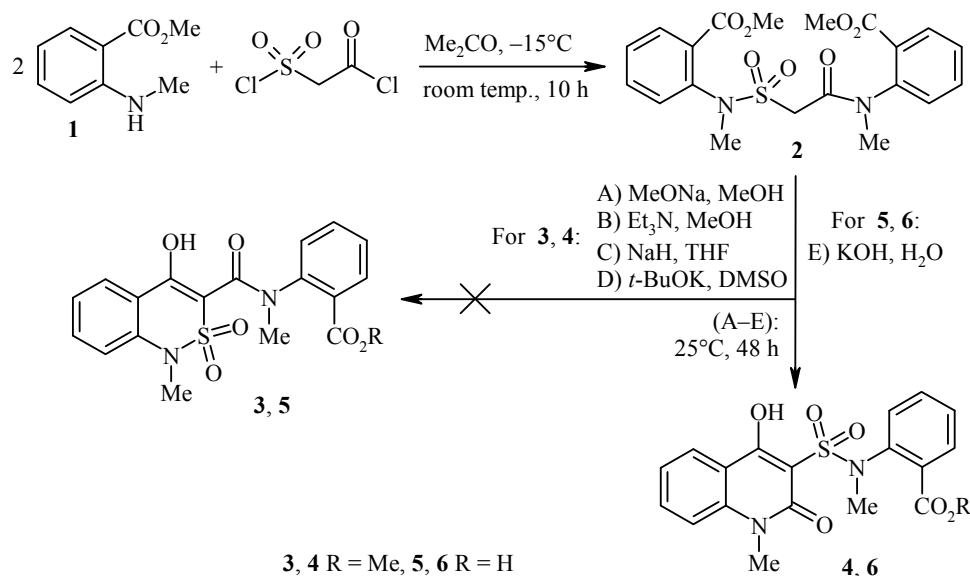
\*For Communication 247, see [1].

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characterization and accurate quantitative determination of all products obtained. For this reason, a complex of modern analytical methods, including the necessary use of HPLC and X-ray structural analysis, has been involved.



A series of intramolecular cyclization experiments was performed with dianilide **2** under anhydrous conditions, using both sodium methoxide in methanol, which is the typical base for Dieckmann condensation (method A), as well as other bases ranging from the relatively weak triethylamine to a strong base, sodium hydride, and a superbases, potassium *tert*-butoxide in DMSO (methods B-G). Regardless of the base selected the similar results were achieved in all of these examples according to HPLC data: the reaction mixtures contained only two compounds – from 96.2 to 99.1% of the cyclic product, and residual dianilide **2**. In other words, heterocyclization of dianilide **2** occurred selectively, and its direction could be determined by the structure of the product obtained.

The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra and mass spectra only allowed to detect the ring closure, but did not reliably differentiate between the possible products – benzothiazine **3** and quinoline **4**, because the spectra did not contradict any of the indicated isomeric structures.

The X-ray structural analysis performed by us clearly solved this analytical problem and allowed to determine that heterocyclization of dianilide **2** under anhydrous conditions occurs exclusively by the pathway of the quinoline ring formation, its sole product is methyl 2-[(4-hydroxy-1-methyl-2-oxo-1,2-dihydroquinolin-3-yl)sulfonyl](methylamino)benzoate (**4**).

The pyridine fragment of the bicyclic system in this molecule (Fig. 1) was slightly non-planar as the maximum value of endocyclic torsion angle  $\text{C}(7)\text{--}\text{C}(8)\text{--}\text{C}(9)\text{--}\text{N}(1)$  was  $-6.8(2)^\circ$ . The sulfanilide substituent at the  $\text{C}(8)$  atom was rotated so that the  $\text{S}(1)\text{=O}(3)$  bond was practically coplanar to the endocyclic double bond  $\text{C}(7)\text{--}\text{C}(8)$  (the torsion angle  $\text{O}(3)\text{--}\text{S}(1)\text{--}\text{C}(8)\text{--}\text{C}(7)$  was  $-11.6(1)^\circ$ ). Such conformation of the sulfanilide substituent was stabilized by the intramolecular hydrogen bond  $\text{O}(2)\text{--}\text{H}\cdots\text{O}(3)$  ( $\text{H}\cdots\text{O}$  1.74 Å,  $\text{O--H}\cdots\text{O}$   $156^\circ$ ), which resulted in lengthening of the  $\text{S}(1)\text{=O}(3)$  bond to 1.451(1) Å and the  $\text{C}(7)\text{--}\text{C}(8)$  bond to 1.372(2) Å, compared to their average values of 1.430 and 1.326 Å, respectively [3], as well as shortening of the  $\text{C}(7)\text{--}\text{O}(2)$  bond to 1.335(2) Å (the average value is 1.362 Å).

The substituent at the sulfo group was in *-ac* conformation relative to the  $\text{C}(7)\text{--}\text{C}(8)$  bond (the torsion angle  $\text{N}(2)\text{--}\text{S}(1)\text{--}\text{C}(8)\text{--}\text{C}(7)$  was  $-125.5(1)^\circ$ ). The  $\text{N}(2)$  atom was practically planar, the sum of its valence angles was  $358.7^\circ$ .

The *N*-methyl group of the anilide fragment was practically orthogonal to the C(8)–S(1) bond (the torsion angle C(8)–S(1)–N(2)–C(11) was  $-101.4(1)^\circ$ ), while a weak intramolecular hydrogen bond C(11)–H(11c)···O(4) was formed (H···O 2.36 Å, C–H···O  $113^\circ$ ). The aromatic ring of this fragment was in *+sc* conformation relative to C(8)–S(1) bond and rotated perpendicular to S(1)–N(2) bond (torsion angles C(8)–S(1)–N(2)–C(12)  $65.2(1)^\circ$ , S(1)–N(2)–C(12)–C(13)  $-96.7(1)^\circ$ ), which was likely caused by repulsion between the aromatic ring and *N*-methyl group (shortened contact H(11a)···C(13) 2.81 Å, while the sum of van der Waals radii [4] was 2.87 Å).

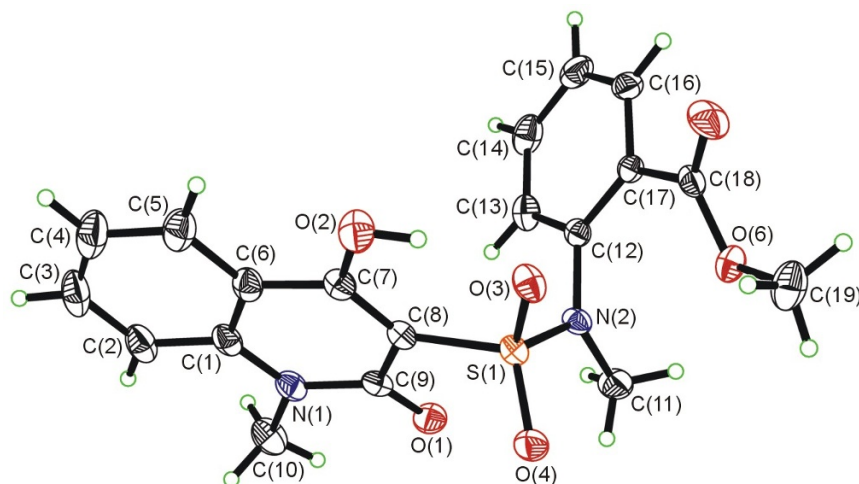


Fig. 1. The molecular structure of ester **4** with atoms represented by thermal vibration ellipsoids of 50% probability.

The ester group was not coplanar with the plane of the aromatic ring (the torsion angle C(16)–C(17)–C(18)–O(5) was  $31.8(2)^\circ$ ), which was apparently a consequence of repulsion between the O(6) and N(2) atoms at 2.77 Å distance, while the sum of van der Waals radii was 2.79 Å. The *O*-methyl group was antiperiplanar to the C(17)–C(18) bond (the torsion angle C(19)–O(6)–C(18)–C(17) was  $-173.8(1)^\circ$ ).

The repulsive interaction between *N*(1)-methyl substituent and atoms of the bicyclic system (shortened intramolecular contacts H(2)···C(10) 2.52 Å (2.87 Å), H(10a)···C(2) 2.83 Å (2.87 Å), H(10b)···O(1) 2.22 Å (2.46 Å), H(10c)···C(2) 2.83 Å (2.87 Å)) led to lengthening of the N(1)–C(9) bond to 1.388(2) Å (1.353 Å) and the N(1)–C(1) bond to 1.388(2) Å (1.371 Å).

The crystal contained an intermolecular hydrogen bond C(15)–H···O(1)' ( $1 + x, y, z$ , H···O 2.29 Å, C–H···O  $156^\circ$ ) between molecules of ester **4**, the formation of which likely led to lengthening of the C(9)–O(1) bond to 1.226(2) Å (the average value is 1.210 Å). Additionally, the crystal contained a system of intermolecular hydrogen bonds C–H··· $\pi$ : C(4)–H···C(15)' ( $\pi$ ) ( $2 -x, 1 -y, 1 -z$ , H··· $\pi$  2.75 Å, C–H··· $\pi$   $145^\circ$ ) and C(4)–H···C(16)' ( $\pi$ ) ( $2 -x, 1 -y, 1 -z$ , H··· $\pi$  2.87 Å, C–H··· $\pi$   $144^\circ$ ).

The use of aqueous KOH solution as a base (method D) did not substantially change the direction of intramolecular heterocyclization of dianilide **2**. The reaction mixture obtained consisted of only two compounds in this case as well: 99.6% of cyclic product with properties different than those of ester **4**, and 0.4% of unreacted starting dianilide **2**. The analysis of  $^1\text{H}$  and  $^{13}\text{C}$  NMR, as well as mass spectra of this sample indicated that the cyclization in this experiment was accompanied by degradation of the ester group and likely formation of acid **6** as an alkaline hydrolysis product of the initially formed methyl ester **4**. The possible formation of isomeric benzothiazine **5** could not be excluded, but was deemed to be less likely when taking into account the results already obtained by methods A–D.

Our assumption regarding acid **6** was completely confirmed by X-ray structural analysis as two molecules of compound **6** (**A** and **B**) in a monohydrate form were found in an independent part of the unit cell. In contrast to methyl ester **4**, the quinolone fragment, carbonyl and hydroxyl groups, as well as the S(1) and O(3) atoms in the molecules **A** and **B** of this compound were located in one plane within 0.02 Å (Fig. 2).

The main differences between molecules **A** and **B** were such that the former contained a substituent at the sulfo group in *-ac* conformation relative to the C(7)–C(8) bond, while the latter – in *+ac* conformation relative to the same bond (the torsion angle N(2)–S(1)–C(8)–C(7) was equal to  $-115.8(2)^\circ$  in molecule **A** and  $119.7(2)^\circ$  in molecule **B**).

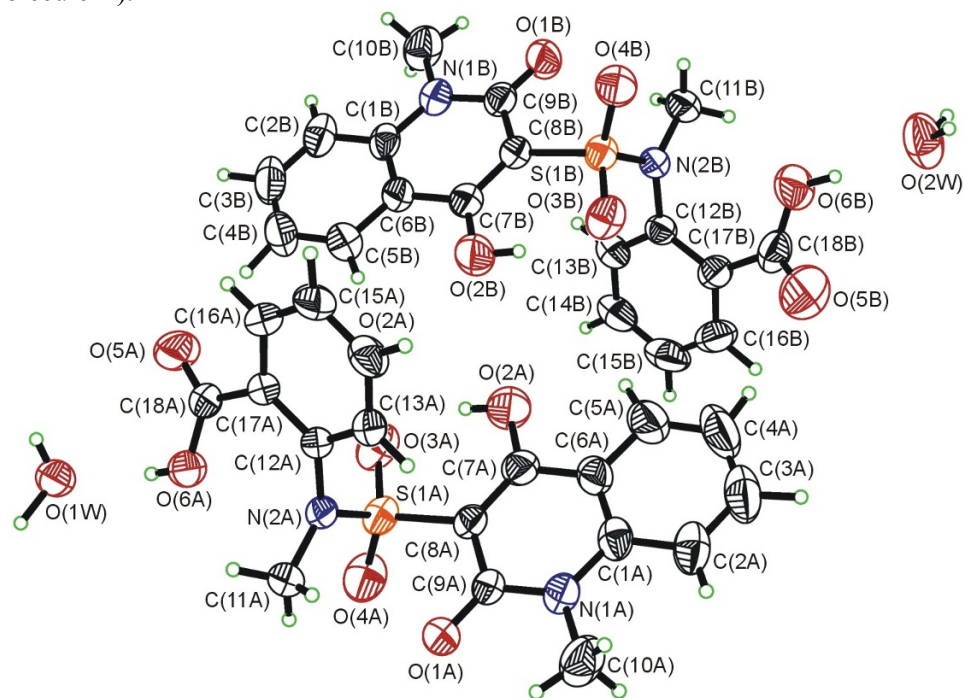


Fig. 2. The structure of carboxylic acid **6** hydrate with atoms represented by thermal vibration ellipsoids of 50% probability.

The methyl group at the N(2) atom was orthogonal to the C(8)–S(1) bond (the torsion angle C(8)–S(1)–N(2)–C(11) was  $-90.2(2)^\circ$  in molecule **A** and  $89.8(2)^\circ$  in molecule **B**), while the aromatic ring was in *+sc* position (molecule **A**) or *-sc* position (molecule **B**) relative to C(8)–S(1) bond and rotated nearly perpendicular to the S(1)–N(2) bond. The carboxyl substituent slightly deviated from the aromatic ring plane, in which it was located (the torsion angle C(16)–C(17)–C(18)–O(5) was  $23.0(3)^\circ$  in molecule **A** and  $-34.2(3)^\circ$  in molecule **B**).

The significant repulsion between the methyl group at the N(1) atom and the adjacent carbonyl group and a hydrogen atom at the *peri* position of the benzene ring, similarly to the case of ester **4**, lengthened the N(1)–C(1) bond to  $1.391(2)$  Å in molecule **A** and  $1.393(2)$  Å in molecule **B**, as well as the N(1)–C(9) bond to  $1.378(2)$  Å in molecule **A** and  $1.379(2)$  Å in molecule **B** compared to the average values of  $1.375$  and  $1.355$  Å, respectively. Apparently, for the same reason, the N(1)-methyl group deviated from the bicyclic fragment plane at angles unusual for quinolones (the torsion angle C(10)–N(1)–C(1)–C(2) was  $5.4(3)^\circ$  in molecule **A** and  $-5.5(3)^\circ$  in molecule **B**). The molecules of acid **6** were linked in a hydrate crystal by several hydrogen bonds through the bridging water molecules.

Thus, our study has clearly shown that despite the superficial structural similarity the closure of 4-hydroxy-2-oxo-1,2-dihydroquinoline ring is energetically more favorable than the closure of 4-hydroxy-2,2-dioxo-1*H*-2λ<sup>6</sup>,1-benzothiazine ring by such margin that only the quinoline system is formed under equivalent conditions. Even though the sulfonyl group confers the hydrogen atoms of neighboring groups with twice as strong acidity compared to the effect of the carbonyl group [5], this obviously facilitates only the initial stage of the cyclization reaction – formation of carbanion. In further stages the larger bulk of the sulfonyl group

as compared to carbonyl hinders the approach of reactive sites and thus substantially interferes with the next stage of the process – the actual ring closure.

## EXPERIMENTAL

$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were acquired on a Varian Mercury-400 instrument (400 and 100 MHz, respectively) in  $\text{DMSO-d}_6$ , with TMS as internal standard. Mass spectra were recorded on a Varian 1200L instrument in a full scan mode over the  $m/z$  range of 35-700, EI ionization (70 eV) with direct introduction of the sample. Elemental analysis was performed on a EuroVector EA-3000 microanalyzer. Melting points were determined in capillary on a Stuart SMP10 digital melting point apparatus. The reaction mixtures formed after treatment of dianilide **2** with bases were analyzed on a modular Bischoff HPLC system with Lambda 1010 spectrophotometric detector (Bischoff Analysentechnik GmbH). The HPLC conditions were: Prontosil 120-5-CN column (4×250 mm); the sorbent particle with the diameter of 5  $\mu\text{m}$ ; the mobile phase flow rate 1 ml/min; the column temperature 40°C; the injection volume 5  $\mu\text{l}$ ; the detector wavelength 254 nm. Mobile phase: 90.5% MeCN and 9.5%  $\text{H}_2\text{O}$ .

**Methyl 2-[(2-{Methyl[2-(methoxycarbonyl)phenyl]amino}-2-oxoethyl)sulfonyl](methylamino)-benzoate (2).** Methyl *N*-methylantranylate (6.77 g, 0.041 mol) (**1**) was added dropwise with vigorous stirring to a solution of chlorosulfonyl acetylchloride (1.77 g, 0.010 mol) in acetone (20 ml) at -15°C. The reaction mixture was left for 10 h at room temperature, then diluted with cold water. The precipitate of dianilide **2** was filtered off, washed with cold water, and dried. Yield 4.05 g (93%), colorless crystals, mp 131-133°C ( $\text{Et}_2\text{O}$ ).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 7.97-7.43 (8H, m, H Ar); 3.82 (2H, s,  $\text{CH}_2$ ); 3.80 (3H, s,  $\text{OCH}_3$ ); 3.76 (3H, s,  $\text{OCH}_3$ ); 3.20 (3H, s,  $\text{NCH}_3$ ); 3.14 (3H, s,  $\text{NCH}_3$ ).  $^{13}\text{C}$  NMR spectrum,  $\delta$ , ppm: 166.8 (COO); 165.7 (COO); 162.1 (CON); 142.6; 140.0; 134.7; 133.7; 132.3; 132.1; 131.2; 131.1; 130.4; 129.8; 129.2; 128.5; 54.2 ( $\text{CH}_2$ ); 53.2 ( $\text{OCH}_3$ ); 52.8 ( $\text{OCH}_3$ ); 37.7 ( $\text{NCH}_3$ ); 37.6 ( $\text{NCH}_3$ ). Mass spectrum,  $m/z$  ( $I_{\text{rel}}$ , %): 435  $[\text{M}+\text{H}]^+$  (2), 370 (39), 190 (25), 165 (79), 164 (64), 147 (19), 133 (37), 132 (100), 118 (18), 105 (62), 104 (57), 77 (75). Found, %: C 55.36; H 5.18; N 6.57; S 7.27.  $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_7\text{S}$ . Calculated, %: C 55.29; H 5.10; N 6.45; S 7.38.

**Methyl 2-[(4-Hydroxy-1-methyl-2-oxo-1,2-dihydroquinolin-3-yl)sulfonyl](methylamino)benzoate (4)** (methods A-D). The corresponding base (0.015 mol) (see scheme) was added to a solution of dianilide **2** (4.34 g, 0.010 mol) in anhydrous MeOH, THF, or DMSO (15 ml). The reaction mixture was stirred for 48 h at 25°C. The mixture was then diluted with cold water and acidified with dilute HCl to pH ~3.0. The precipitate of ester **4** obtained was filtered off, washed with cold water, and dried. Yields 3.54-3.78 g (88-94%), colorless crystals, mp 180-182°C (DMF).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 11.00 (1H, br. s, 4-OH); 7.91 (1H, dd,  $^3J = 8.0$ ,  $^2J = 1.5$ , H-5); 7.79 (1H, td,  $^3J = 7.7$ ,  $^2J = 1.5$ , H-7); 7.73 (1H, dd,  $^3J = 7.0$ ,  $^2J = 2.2$ , H-3'); 7.59 (1H, d,  $J = 8.5$ , H-8); 7.50 (1H, td,  $^3J = 7.6$ ,  $^2J = 2.2$ , H-5'); 7.43 (1H, td,  $^3J = 7.6$ ,  $^2J = 2.2$ , H-4'); 7.30 (1H, t,  $J = 7.6$ , H-6); 7.22 (1H, dd,  $^3J = 6.7$ ,  $^2J = 2.2$ , H-6'); 3.78 (3H, s,  $\text{OCH}_3$ ); 3.63 (3H, s,  $\text{NCH}_3$ ); 3.49 (3H, s,  $\text{NCH}_3$ ).  $^{13}\text{C}$  NMR spectrum,  $\delta$ , ppm: 166.7 (COO); 163.2 (2-CO); 158.4 (C-4); 140.9; 138.9; 135.2; 133.8; 131.1; 130.3; 129.7; 125.4; 123.1; 121.9; 115.8; 114.8; 111.7; 52.8 ( $\text{OCH}_3$ ); 42.1 ( $\text{NCH}_3$ ); 29.7 ( $\text{NCH}_3$ ). Mass spectrum,  $m/z$  ( $I_{\text{rel}}$ , %): 402  $[\text{M}]^+$  (1), 371  $[\text{M}-\text{MeOH}]^+$  (15), 211 (46), 175 (22), 165 (68), 164 (90), 149 (11), 147 (17), 146 (89), 133 (39), 132 (100), 118 (25), 105 (65), 104 (67), 91 (58), 86 (49). Found, %: C 56.65; H 4.58; N 7.03; S 7.88.  $\text{C}_{19}\text{H}_{18}\text{N}_2\text{O}_6\text{S}$ . Calculated, %: C 56.71; H 4.51; N 6.96; S 7.97.

**2-[(4-Hydroxy-1-methyl-2-oxo-1,2-dihydroquinolin-3-yl)sulfonyl](methylamino)benzoic Acid Hydrate (6)** (method E). Obtained from dianilide **2** according to the previous procedure, the solvent was  $\text{H}_2\text{O}$ , the basic catalyst was KOH. Yield 90%, colorless crystals, mp 157-159°C (DMF).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 11.02 (1H, br. s, 4-OH); 7.90 (1H, dd,  $^3J = 8.1$ ,  $^2J = 1.5$ , H-5); 7.83-7.74 (2H, m, H-7,3'); 7.60 (1H, d,  $J = 8.5$ , H-8); 7.48-7.38 (2H, m, H-4',5'); 7.30 (1H, t,  $J = 7.6$ , H-6); 7.13 (1H, dd,  $^3J = 6.6$ ,  $^2J = 2.2$ , H-6'); 3.63 (3H, s,  $\text{NCH}_3$ ); 3.50 (3H, s,  $\text{NCH}_3$ ).  $^{13}\text{C}$  NMR spectrum,  $\delta$ , ppm: 167.8 (COOH); 163.2 (2-C=O); 158.6 (C-4); 141.1; 138.9; 135.2; 133.4; 132.3; 130.1; 129.7; 125.0; 123.1; 122.0; 115.9; 114.9; 112.4; 42.4 ( $\text{NCH}_3$ ); 29.8 ( $\text{NCH}_3$ ). Mass

spectrum,  $m/z$  ( $I_{\text{rel}}$ , %): 306 (3), 175 (100), 151 (65), 150 (36), 149 (22), 147 (12), 146 (35), 133 (27), 132 (41), 117 (15), 105 (83), 104 (98), 91 (13), 77 (71). Found, %: C 53.28; H 4.52; N 6.80; S 7.83.  $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_6\text{S}\cdot\text{H}_2\text{O}$ . Calculated, %: C 53.20; H 4.46; N 6.89; S 7.89.

**X-Ray Structural Investigation of Methyl Ester 4.** Crystals of ester **4** ( $\text{C}_{19}\text{H}_{18}\text{N}_2\text{O}_6\text{S}$ ,  $M$  402.41) were monoclinic (DMF), at  $-153^\circ\text{C}$ :  $a$  8.7822(4),  $b$  15.9726(7),  $c$  13.0922(6) Å;  $\beta$  92.286(1) $^\circ$ ;  $V$  1835.0(1) Å<sup>3</sup>;  $Z$  4; the space group  $P2_1/c$ ;  $d_{\text{calc}}$  1.457 g/cm<sup>3</sup>;  $\mu(\text{MoK}\alpha)$  0.217 mm<sup>-1</sup>,  $F(000)$  840. Unit cell parameters and intensities of 23374 reflections (5366 independent,  $R_{\text{int}}$  0.040) were measured on a Bruker-Apex2 diffractometer (MoK $\alpha$  radiation, CCD-detector, graphite monochromator,  $\omega$ -scanning,  $2\theta_{\text{max}}$  60 $^\circ$ ). The structure was solved by the direct method using the SHELXTL software suite [6]. The hydrogen atom positions were revealed from differential synthesis of electron density and refined in isotropic approximation. The structure was refined by  $F^2$  with the full matrix method of least squares in anisotropic approximation for non-hydrogen atoms to  $wR_2$  0.104 by 5339 reflections ( $R_1$  0.042 by 4166 reflections with  $F > 4\sigma(F)$ ,  $S$  1.024). The complete crystallographic data set for methyl ester **4** was deposited at the Cambridge Crystallographic Data Center (deposit CCDC 1010032).

**X-Ray Structural Study of Carboxylic Acid 6 Hydrate.** Crystals of carboxylic acid **6** hydrate ( $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_6\text{S}\cdot\text{H}_2\text{O}$ ,  $M$  406.40) were monoclinic (DMF), at  $20^\circ\text{C}$ :  $a$  13.0490(5),  $b$  10.9998(3),  $c$  25.948(1) Å;  $\beta$  91.833(3) $^\circ$ ;  $V$  3722.5(2) Å<sup>3</sup>;  $Z$  4, the space group  $P2_1/c$ ;  $d_{\text{calc}}$  1.450 g/cm<sup>3</sup>;  $\mu(\text{MoK}\alpha)$  0.219 mm<sup>-1</sup>;  $F(000)$  1696. The unit cell parameters and intensities of 42895 reflections (10841 independent,  $R_{\text{int}}$  0.037) were measured on an Xcalibur-3 diffractometer (MoK $\alpha$  radiation, CCD-detector, graphite monochromator,  $\omega$ -scanning,  $2\theta_{\text{max}}$  60 $^\circ$ ). The structure was solved by the direct method using the SHELXTL software suite [6]. The hydrogen atom positions were revealed from differential synthesis of electron density and refined in isotropic approximation. The structure was refined by  $F^2$  with the full matrix method of least squares in anisotropic approximation for non-hydrogen atoms to  $wR_2$  0.139 by 10799 reflections ( $R_1$  0.051 by 6516 reflections with  $F > 4\sigma(F)$ ,  $S$  0.999). The complete crystallographic data set for hydrate of the acid **6** was deposited at the Cambridge Crystallographic Data Center (deposit CCDC 1010031).

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