Unusual reaction of 2-(trifluoromethyl)-1,2-dihydro-3 λ^6 -thieno[2,3-*c*]chromen-3,3,4-triones with hydrazine as a new route to 3-hydrazinopyridazine derivatives

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Discussion and X-ray data for compound 5b

In the Cambridge Structural Database (CSD),¹ we found 3 structures containing the pyridazinium fragment with the nitrogen atom (analog of N(3)) and phenyl substituent in 6- and 3-positions of the cycle as in cation **5b**. These are *N*-(2-morpholin-4-ylethyl)-6-phenylpyridazin-3-amine dihydrochloride monohydrate **7**,² *N*-(2-morpholin-4-ylethyl)-4,6-diphenylpyridazin-3-amine dihydrochloride tetrahydrate **8**,³ and 5-methyl-*N*-(2-morpholin-4-ylethyl)-6-phenylpyridazin-3-amine oxalate oxalic acid **9**⁴ (Fig. S1). The geometric parameters of the fragments we are interested in are presented in Table 1. As can be seen from these data, the character of distribution of the bond lengths in the C(5)–C(4)=N(1)–N(2)=C(1)–N(3) chain is similar in all structures **5b**, **7-9**, and in structures **5b** and **7** the geometry of the whole specified fragment is in fact identical. Thus, the bond length distribution in the heterocycle and hydrazine fragment found for cation **5b** is characteristic, most likely, for this class of compounds.

Figure S1

It is of interest to compare the bond distribution in cation **5b**, 1-(*tert*-butylamino)-3-[2-(6-hydrazinopyridazin-3-yl)phenoxy]propan-2-ol hemisulfate monohydrate (prizidilol) **10** (Table S1), and pyridazine.⁵ Compound **10** contains as a fragment the analog of cation **5b** (without Me group), which

deprotonated over the N(2) atom (neutral) and whose phenol group is not conjugated with the pyridazine ring (the angle between the planes is $\sim 48^\circ$) and NHNH_2 group is antiperiplanar with respect to the N(2) atom. In cation **5b**, the $\text{C}(2)=\text{C}(3)$ bond of 1.342(3) Å is localized to a greater extent than those in **10** (1.356 Å) and pyridazine (1.371 Å), whereas the $\text{C}(1)-\text{C}(2)$ 1.421(3) and $\text{C}(3)-\text{C}(4)$ 1.437(3) Å bond lengths are longer than those in **10** (1.398 and 1.417 Å, respectively) and pyridazine (average 1.393 Å), and the $\text{N}(1)=\text{C}(4)$ 1.318(3) bond is, in fact, the same (average 1.325 Å for **10** and pyridazine). The $\text{N}(1)-\text{N}(2)$ 1.356(2) and $\text{N}(2)=\text{C}(1)$ 1.333(3) Å bonds are close to analogous bonds in pyridazine (1.346 Å for $=\text{N}-\text{N}=-$ and 1.327 Å on the average for $-\text{N}=\text{C}-$) but noticeably differ from those in molecule **10** (1.335 and 1.352 Å, respectively). Note also the strengthening of the $\text{C}(1)-\text{N}(3)$ 1.316(3) Å bond in cation **5b** compared to the analogous one in **10** (1.349 Å). Thus, in the pyridazinium fragment of cation **5b** the bonds are predominantly localized except for the $\text{N}(2)=\text{C}(1)-\text{N}(3)$ chain in which the strong electron density delocalization is observed. This can be related to the presence of the hydrogen on the N(2) atom, the conjugation in the $\text{C}(5)-\text{C}(4)=\text{N}(1)-\text{N}(2)=\text{C}(1)-\text{N}(3)$, and the intramolecular interaction of the electron pair of the N(4) with the proton on the N(2) atom.

The $\text{N}(4)\text{H}_2$ amino group is periplanar with respect to the N(2) nitrogen atom. Analysis (performed using CSD) of the geometry of the $\text{HN}(2)=\text{C}(1)-\text{N}(3)\text{H}-\text{N}(4)\text{H}_2$ fragment in amino-substituted guanidinium species showed that the conformation observed in **5b** (the N(2) and N(4) atoms in the periplanar position, the hydrogen atoms of the pyramidal N(4) nitrogen atoms are oriented to the side opposite from the N(2)H group) takes place in 11 of 12 found structures and it seems to be rather stable. Parameters of $\text{N}-\text{H}\cdots\text{NH}_2$ contact lie in the following intervals: $\text{H}\cdots\text{N}$ 2.19–2.40 (average 2.33 Å), $\text{N}\cdots\text{N}$ 2.618–2.711 (average 2.674 Å), angle $\text{N}-\text{H}\cdots\text{N}$ 99–111 (average 104°). Perhaps, the $\text{N}(2)-\text{H}(2\text{N})\cdots\text{N}(4)$ contact in **5b** (distances $\text{H}\cdots\text{N}$ 2.34(3), $\text{N}\cdots\text{N}$ 2.660(3) Å, angle $\text{N}-\text{H}\cdots\text{N}$ $106(3)^\circ$) also corresponds to some attractive interaction (intramolecular hydrogen bond), which stabilizes the observed conformation of the hydrazine fragment.

Another factor favoring the electron density delocalization and flattening of the molecule is the stacking interaction by which the molecules form center-symmetric dimers in crystal **5b** (Fig. 2). The distance between the medium planes of the cations is 3.371 Å. The shortest distances between the atoms of the dimer arranged one above another and between the carbon atoms and centers of the pyridazinium and phenol cycles are presented in Table S2. Note that the pyridazinium and hydrazine fragments arranged one above another are not absolutely parallel to each other. The angle between their planes is 8.2° that leads to the different intermolecular distances in the dimer. The very short C(2)...C(6)^{#4} 3.154(3) Å distance (Table S2) can be explained, most likely, by some excess of the π -density on the C(2) atom due to the earlier mentioned high localization of the C(2)=C(3) bond and its deficient on the C(6) atom caused by the influence of the hydroxyl group. Thus, the stacking interaction in structure **5b** can be accompanied by charge transfer.

The environment of the Cl atom of the perchlorate anion is close to tetrahedral (angles O–Cl–O 108.4(1)–110.6(1)°). The Cl–O bond lengths change rather substantially (1.427(2)–1.454(2) Å), that may be due to the difference in intermolecular interactions of oxygen atoms (Table S2). It is seen in Fig. 2 that the perchlorate anions and dimer cations interact with each other by the formation of a branched network of hydrogen bonds. Thus, virtually all functional groups of the cation and the oxygen atoms of the perchlorate anion are involved in the system of intra- and intermolecular hydrogen bonds. This can play a considerable role in the formation of the complicated bond length distribution in both the cation and anion in the structure **5b**.

- (1) Cambridge Structural Database, Release **2002**, October.
- (2) Michel, A.; Gustin, R.; Evrard, G.; Durant, F. *Bull. Soc. Chim. Belg.* **1982**, *91*, 49.
- (3) Michel, A.; Gustin, R.; Evrard, G.; Durant, F. *Bull. Soc. Chim. Belg.* **1982**, *91*, 123.
- (4) Van der Brempt, C.; Durant, F.; Norberg, B.; Evrard, G. *Acta Crystallogr.* **1985**, *C41*, 1532.
- (5) Egli, M.; Dobler, M.; *Helv. Chim. Acta* **1989**, *72*, 1136.

Table S1. Selected bond lengths (*d*/Å) and angles (*ω*/deg.) in compounds **5b**, **7-10**

Code of CSD		BAFJEX10	BEXVIJ	DEMYAV	HAVHIT
	5b	7	8	9	10
N(1)-C(4)	1.318(3)	1.308	1.320	1.319	1.323
N(1)-N(2)	1.356(2)	1.364	1.364	1.345	1.335
N(2)-C(1)	1.333(3)	1.336	1.336	1.351	1.352
C(1)-C(2)	1.421(3)	1.423	1.455	1.428	1.398
C(2)-C(3)	1.342(3)	1.346	1.354	1.341	1.356
C(3)-C(4)	1.437(3)	1.429	1.417	1.441	1.417
N(3)-C(1)	1.316(3)	1.323	1.340	1.317	1.349
N(3)-N(4)	1.413(3)				1.404
C(4)-C(5)	1.479(3)	1.491	1.477	1.490	1.469
C(5)-C(6)	1.412(3)				1.385
O(1)-C(6)	1.364(3)				
C(5)-C(10)	1.402(3)				1.396
C(6)-C(7)	1.391(3)				
C(9)-C(11)	1.515(3)				
C(4)-N(1)-N(2)	118.0(2)	116.898	117.264	117.425	122.1
C(1)-N(2)-N(1)	125.6(2)	126.014	126.103	125.775	119.2
N(2)-C(1)-C(2)	117.1(2)	116.704	117.151	116.359	121.7
C(3)-C(2)-C(1)	118.9(2)	118.991	116.524	120.049	117.9
C(2)-C(3)-C(4)	120.2(2)	119.625	122.031	118.478	119.0
N(1)-C(4)-C(3)	120.2(2)	121.704	120.782	121.862	120.0
N(3)-C(1)-N(2)	120.1(2)	121.354	121.527	117.690	113.3
C(1)-N(3)-N(4)	118.5(2)				123.8
N(1)-C(4)-C(5)	117.3(2)	116.029	115.914	115.821	115.5
C(6)-C(5)-C(4)	122.9(2)	116.898	117.264	117.425	122.5
O(1)-C(6)-C(5)	124.1(2)				
C(3)-C(4)-C(5)	122.5(2)				124.5
N(3)-C(1)-C(2)	122.7(2)				125.1

C(10)-C(5)-C(6)	117.3(2)
C(7)-C(6)-C(5)	119.7(2)

Table S2. Parameters of intermolecular hydrogen bonds and contacts (distances d and angles ω) and some intermolecular distances in structure **5b** [$\text{\AA}$ and deg.]. X1A and X1B are the geometric centers of benzene and pyridazine cycles, respectively.

D-H...A	$d(\text{D-H})$	$d(\text{H...A})$	$d(\text{D...A})$	$\omega(\text{DHA})$
O(1)-H(1O)...N(1)	0.88(3)	1.88(4)	2.653(2)	146(3)
N(2)-H(2N)...N(4)	0.77(3)	2.34(3)	2.660(3)	106(3)
O(1)-H(1O)...O(1S)#1	0.88(3)	2.32(4)	2.756(2)	111(3)
N(2)-H(2N)...O(3S)#1	0.77(3)	2.24(3)	2.960(3)	156(3)
N(3)-H(3N)...O(1S)	0.85(3)	2.01(3)	2.854(3)	177(2)
N(4)-H(4NA)...O(2S)#2	0.90(3)	2.28(4)	3.068(3)	145(3)
N(4)-H(4NB)...O(3S)#3	0.94(3)	2.35(3)	3.167(3)	146(2)
C(2)...C(6)#4			3.154(3)	
C(4)...C(4)#4			3.329(4)	
X(1A)...C(5)#4			3.333(3)	
X(1B)...C(1)#4			3.341(3)	
N(3)...C(8)#4			3.379(3)	
N(2)...C(10)#4			3.486(3)	

Symmetry transformations used to generate equivalent atoms: #1 $x, y, z-1$; #2 $-x, y, -z+3/2$; #3 $x, -y+1, z-1/2$; #4 $1/2-x, 1/2-y, 1-z$.

Complete listing of IR and ¹H NMR peaks and elemental analyses of compounds 3d-3h, 4b, 5b and

¹³C NMR peaks for 4b

7,9-Dimethyl-2-(trifluoromethyl)-1,2-dihydro-3λ⁶-thieno[2,3-*c*]chromen-3,3,4-trione (3d): yield 71%; mp 237–238 °C; ¹H NMR (DMSO-*d*₆/CCl₄) δ 2.44 (s, 3H, Me), 2.72 (s, 3H), 3.85 (dd, 1H, *J* = 18.3, 6.1 Hz), 4.25 (dd, 1H, *J* = 18.3, 9.0 Hz), 4.90–4.97 (m, 1H), 7.10 (d, 1H, *J* = 1.5 Hz), 7.18 (d, 1H, *J* = 1.5 Hz). IR (Nujol) 1730 (C=O), 1625, 1600, 1545 cm⁻¹. Anal. Calcd for C₁₄H₁₁F₃O₄S: C, 50.60; H, 3.34. Found: C, 50.80; H, 3.46.

2-(Trifluoromethyl)-1,2-dihydro-3λ⁶-benzo[*f*]thieno[2,3-*c*]chromen-3,3,4-trione (3e): yield 63%; mp 300–301 °C (DMSO–EtOH, 1:1); ¹H NMR (DMSO-*d*₆) δ 4.34 (dd, 1H, *J* = 17.9, 6.3 Hz), 4.50 (dd, 1H, *J* = 17.9, 8.9 Hz), 5.20–5.30 (m, 1H), 7.70 (d, 1H, *J* = 9.0 Hz), 7.72 (t, 1H, *J* = 7.2 Hz), 7.84 (ddd, 1H, *J* = 8.6, 7.0, 1.5 Hz), 8.16 (dd, 1H, *J* = 8.1, 1.3 Hz), 8.44 (d, 1H, *J* = 9.0 Hz), 8.53 (d, 1H, *J* = 8.7 Hz); IR (Nujol) 1745 (C=O), 1620, 1600, 1580, 1545, 1515 cm⁻¹. Anal. Calcd for C₁₆H₉F₃O₄S: C, 54.24; H, 2.56. Found: C, 54.37; H, 2.73.

2-(Trifluoromethyl)-2,3-dihydro-1λ⁶-benzo[*h*]thieno[2,3-*c*]chromen-1,1,11-trione (3f): yield 48%; mp 314–315 °C (DMSO–EtOH, 1:1); ¹H NMR (DMSO-*d*₆) δ 3.95 (dd, 1H, *J* = 19.0, 5.9 Hz), 4.20 (dd, 1H, *J* = 19.0, 9.1 Hz), 5.31–5.41 (m, 1H), 7.78 (ddd, 1H, *J* = 8.2, 7.0, 1.3 Hz), 7.85 (ddd, 1H, *J* = 8.2, 7.0, 1.3 Hz), 7.94 (d, 1H, *J* = 8.7 Hz), 7.99 (d, 1H, *J* = 8.7 Hz), 8.11 (d, 1H, *J* = 7.9 Hz), 8.39 (d, 1H, *J* = 8.3 Hz); IR (Nujol) 1745 (C=O), 1610, 1560 cm⁻¹. Anal. Calcd for C₁₆H₉F₃O₄S: C, 54.24; H, 2.56. Found: C, 54.46; H, 2.65.

8-Nitro-2-(trifluoromethyl)-1,2-dihydro-3λ⁶-thieno[2,3-*c*]chromen-3,3,4-trione (3g). Sulfone **3a** (1.0 g, 3.3 mmol) was dissolved in concentrated H₂SO₄ (3 mL), and a mixture of concentrated H₂SO₄ (1 mL) and anhydrous HNO₃ (0.5 mL) was added with stirring to the resulting solution. The reaction mixture was left to stand at ~20 °C for 10 min and then mixed with ice-cold water (100 mL). The precipitate that formed was filtered off, washed with water, and dried to give a colorless solid **3g** (1.08

g, 94%), mp 315–316 °C (decomp.). ^1H NMR (DMSO- d_6) δ 4.05 (dd, 1H, J = 19.4, 5.6 Hz), 4.24 (dd, 1H, J = 19.4, 9.1 Hz), 5.34–5.44 (m, 1H, CH), 7.81 (d, 1H, J = 9.2 Hz), 8.64 (dd, 1H, J = 9.2, 2.7 Hz), 8.93 (d, 1H, J = 2.7 Hz); IR (Nujol) 3105 (=CH), 1750 (C=O), 1630, 1580, 1530 cm^{-1} . Anal. Calcd for $\text{C}_{12}\text{H}_6\text{F}_3\text{NO}_6\text{S}$: C, 41.27; H, 1.73; N, 4.01. Found: C, 41.19; H, 1.65; N, 4.16.

8-Amino-2-(trifluoromethyl)-1,2-dihydro-3 λ^6 -thieno[2,3-*c*]chromen-3,3,4-trione (3h). Ethanol saturated with HCl (6 mL) and nitrosulfone **3g** (1.0 g, 2.9 mmol) were added to a solution of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ (2.8 g, 12.4 mmol) in ethanol (15 mL). The reaction mixture was refluxed for 10 min until a homogeneous solution was formed and then cooled to room temperature, after which pyridine (20 mL) was added. The precipitate that formed, being a mixture of aminosulfone **3h** and inorganic tin compounds, was filtered off, washed with water, dried, and mixed with acetone (100 mL). The obtained extract was filtered, the precipitate was washed with acetone (100 mL), acetone excess was distilled off, and the residue was cooled to -10 °C. The yellow crystals that precipitated were filtered off, washed with cooled acetone (5 mL), and dried to give **3h** (0.60 g, 66%), mp 244–245 °C (decomp.). ^1H NMR (DMSO- d_6) δ 3.75 (dd, 1H, J = 18.6, 6.0 Hz), 3.94 (dd, 1H, J = 18.6, 9.1 Hz), 5.15–5.25 (m, 1H), 5.54 (s, 2H), 6.95 (d, 1H, J = 2.7 Hz), 7.11 (dd, 1H, J = 8.9, 2.7 Hz), 7.31 (d, 1H, J = 8.9 Hz); IR (Nujol) 3460, 3370, 3250 (NH_2), 1730, 1715 (C=O), 1645, 1630, 1580, 1500 cm^{-1} . Anal. Calcd for $\text{C}_{12}\text{H}_8\text{F}_3\text{NO}_4\text{S}$: C, 45.15; H, 2.53; N, 4.39. Found: C, 45.04; H, 2.47; N, 4.36.

2-(6-Hydrazinopyridazin-3-yl)-4-methylphenol monohydrate (4b): yield 74%; mp 203–204 °C as a colorless needles from ethanol; ^1H NMR (DMSO- d_6) δ 2.29 (s, 3H), 4.44 (br s, 2H), 6.83 (d, 1H, J = 8.3 Hz), 7.07 (dd, 1H, J = 8.3, 1.7 Hz), 7.26 (d, 1H, J = 9.7 Hz), 7.64 (d, 1H, J = 1.5 Hz), 8.16 (d, 1H, J = 9.7 Hz), 8.32 (br s, 1H), 13.50 (br s, 1H); ^{13}C NMR (DMSO- d_6) δ 20.16 (qt, Me, 1J = 126.1, 3J = 4.4 Hz), 115.13 (d, $\text{C}^{5'}$, 1J = 169.6 Hz), 117.15 (d, C^6 , 1J = 156.9 Hz), 117.36 (d, C^2 , 3J = 5.2 Hz), 125.13 (d, $\text{C}^{4'}$, 1J = 167.3 Hz), 125.88 (ddq, C^3 , 1J = 155.2, $^3J_{\text{C}^3,\text{H}^5}$ = 6.6, $^3J_{\text{C}^3,\text{Me}}$ = 5.2 Hz), 127.19 (dq, C^4 , $^3J_{\text{C}^4,\text{H}^6}$ = 7.6, $^2J_{\text{C}^4,\text{Me}}$ = 6.1 Hz), 130.71 (ddq, C^5 , 1J = 156.6, $^3J_{\text{C}^5,\text{H}^3}$ = 7.7, $^3J_{\text{C}^5,\text{Me}}$ = 4.6 Hz), 152.12

(br s, C⁶), 155.75 (t, C¹, ³*J* = 8.9 Hz), 160.49 (t, C^{3'}, ³*J* = 6.6 Hz), the assignment of the protonated carbon atoms was confirmed by the data of the 2D-HETCOR spectrum; IR (Nujol) 3360, 3330, 3180–3300, 1620, 1600 cm⁻¹. Anal. Calcd for C₁₁H₁₂N₄O·H₂O: C, 56.40; H, 6.02; N, 23.92. Found: C, 56.39; H, 5.66; N, 23.91.

3-Hydrazino-6-(2-hydroxy-5-methylphenyl)pyridazin-2-ium perchlorate (5b). Pyridazine **4b** (100 mg, 0.43 mmol) was mixed with water (3 mL), 3 drops of concentrated HCl were added, the mixture was heated to boiling, and 3 drops of concentrated HClO₄ were added. After the resulting solution was cooled, a yellow crystals were crystallized, filtered off, washed with a small amount of water and ethanol, and dried to give perchlorate **5b** (0.12 g, 89%), mp 265–267 °C. ¹H NMR (DMSO-d₆) δ 2.28 (s, 3H), 3.43 (br s, 5H), 6.90 (d, 1H, *J* = 8.3 Hz), 7.16 (dd, 1H, *J* = 8.3, 1.6 Hz), 7.41 (d, 1H, *J* = 9.6 Hz), 7.54 (s, 1H), 8.28 (d, 1H, *J* = 9.6 Hz), 10.06 (br s, 1H), 11.3 (br s, 1H); IR (Nujol) 3385, 3200, 1690, 1570 cm⁻¹. Anal. Calcd for C₁₁H₁₂N₄O·HClO₄: C, 41.72; H, 4.14; N, 17.69. Found: C, 41.73; H, 4.10; N, 17.77.

S10

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LB1 = 0.20 Hz

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PTSld = 32768

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SW1 = 4006.41 Hz

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O1 = 1599.42 Hz

LB1 = 0.20 Hz

TP A = -21.00

B = 0.00

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F1 = 400.133209 MHz

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LB1 = 0.20 Hz

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B = -211.30

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SW1 = 7183.91 Hz

AT1 = 4.56 sec

Hz per Pt 1stD = 0.22 Hz

O1 = 3401.81 Hz

LB1 = 0.20 Hz

TP A = 94.26

B = -207.41

4g

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PTSID = 32768

F1 = 400.132416 MHz

SW1 = 5592.84 Hz

AT1 = 5.86 sec

Harper P1 std = 0.17 Hz

O1 = 2399.70 Hz

LB1 = 0.20 Hz

TP A = 119.50

B = 0.00

6g

1.00

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Wed Nov 21 10:46:57 2001

USER:

Experiment = 2030

Pulse length = 10.10 μ sec

Recycle delay = 1.00 sec

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PTSID = 32768

F1 = 400.132416 MHz

$$SW1 = 5592.84 \text{ Hz}$$
$$AT1 = 5.86 \text{ sec}$$
$$\text{Hz per Pt 1stD} = 0.17 \text{ Hz}$$
$$f_{01} = 2402.35 \text{ Hz}$$
$$LB1 = 0.20 \quad Hz$$

TP A = 48.00

$$B = 0.00$$

75% 4g

25% 6g

TISER.

$$B = 0.00$$