Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Synthesis, Conformation and Antiproliferative Activity of Isothiazoloisoxazole 1,1-dioxides

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ELECTRONIC SUPPLEMENTARY INFORMATION FILE 3:

Crystallographic data summary for compounds **22a, 22h (both isomers), 22i, 24 and 23b**

Crystallographic Data						
	Compounds					
Data	**22a**	**22h-D1**	**22h-D2**	**22i**	**24**	**23b**
Empirical formula	$C_{18}H_{23}N_3O_6S$	$C_{23}H_{27}N_3O_6S_2$	$C_{23}H_{27}N_3O_6S_2$	$C_{23}H_{27}N_3O_7S_2$	$C_{27}H_{28}N_4O_3S$	$C_{15}H_{21}N_5O_4S_2$
Molecular weight (g/mol)	409.45	505.60	505.60	521.60	488.59	399.48
Temperature (K)	120(2)	100(2)	100(2)	100(2)	120(2)	100(2)
Wavelength (Å)	0.71073	0.71073	0.71073	0.71073	0.71073	0.71073
Crystal system	Orthorhombic	Triclinic	Monoclinic	Triclinic	Monoclinic	Monoclinic
Space group	Pbca	P-1	P2(1)/n	P-1	P2(1)/c	P2(1)/n
Unit cell dimensions						
a (Å)	12.9462(7)	8.9873(5)	11.789(1)	8.9413(4)	8.9260(8)	9.3651(3)
b (Å	14.7915(7)	11.0990(6)	14.752(2)	11.7201(5)	15.4665(7)	19.5330(6)
c (Å)	21.2167(8)	13.0584(7)	14.574(2)	13.5584(6)	17.789(1)	12.7944(4)
α (˚)	90.00	70.595(1)	90.00	95.727(1)	90.00	90.00
β (˚)	90.00	76.291(1)	113.86	94.027(1)	99.900(3)	109.830(1)
γ (˚)	90.00	83.975(2)	90.00	105.992(1)	90.00	90.00
Volume (Å^3)	4062.9(3)	1193.1(1)	2318.0(6)	1351.9(1)	2419.3(3)	2201.6(1)
Z	8	2	4	4	4	4
Density (calculated) (mg/m^3)	1.339	1.491	1.42	1.352	1.341	1.272
Absorption coefficient (mm^{-1})	0.198	0.276	0.274	0.429	0.171	0.272
Crystal	cut block, colourless	cut block, colourless	cut block, colourless	cut block, colourless	cut block, colourless	cut block, colourless
θ range for data collection (˚)	3.15-27.48	1.69-35.63	2.06-28.48	1.82-29.63	2.99-27.21	1.99-29.22
Index ranges	$-14 \le h \le 16$ $-19 \le k \le 19$ $-27 \le l \le 27$	$-14 \le h \le 14$ $-18 \le k \le 18$ $-21 \le l \le 21$	$-15 \le h \le 15$ $-19 \le k \le 18$ $-19 \le l \le 8$	$-12 \le h \le 12$ $-16 \le k \le 16$ $-18 \le l \le 12$	$-11 \le h \le 11$ $-19 \le k \le 18$ $-22 \le l \le 22$	$-12 \le h \le 12$ $-26 \le k \le 26$ $-17 \le l \le 17$
Reflections collected	26596	43008	15611	28474	31632	23543
Independent collections	4656 R_{int} = 0.0769	10964 R_{int} = 0.0348	4259 R_{int} = 0.0702	7503 R_{int} = 0.0258	5360 R_{int} = 0.1765	5953 R_{int} = 0.0357
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	4656 / 0 / 258	10964 / 0 / 312	4259 / 0 / 312	7503 / 0 / 348	5360 / 0 /316	5953 / 0 / 281
Goodness-of-fit on F^2	1.054	1.013	0.760	1.028	1.010	0.970
Final R indices [$F^2 > 2\sigma(F^2)$]	$R1$ = 0.0565 $wR2$ = 0.1281	$R1$ = 0.0379 $wR2$ = 0.1026	$R1$ = 0.0402 $wR2$ = 0.0822	$R1$ = 0.0307 $wR2$ = 0.0793	$R1$ = 0.0734 $wR2$ = 0.1418	$R1$ = 0.0321 $wR2$ = 0.0813
R indices (all data)	$R1$ = 0.0982 $wR2$ = 0.1488	$R1$ = 0.0502 $wR2$ = 0.1100	$R1$ = 0.0769 $wR2$ = 0.0916	$R1$ = 0.0358 $wR2$ = 0.0828	$R1$ = 0.1647 $wR2$ = 0.1645	$R1$ = 0.0432 $wR2$ = 0.0874
Extinction coefficient	0.0017(5)	-	-	-	-	-
Largest diff. peak and hole (e/Å^3)	0.755 and -0.363	1.579 and -0.309	0.256 and -0.502	0.447 and -0.508	0.481 and -0.481	0.519 and -0.397

Suitable crystals for (**22a**) and (**24**) were selected and data collected on a Bruker Nonius KappaCCD Area Detector at the window of a Bruker Nonius FR591 rotating anode ($\lambda_{Mo\text{-}K\alpha}$ = 0.71073 Å) driven by COLLECT[i] and DENZO[ii] software at 120 K. The structure was determined in SHELXS-97[iii] and refined using SHELXL-2014.[iv] CCDC1441502 and CCDC1441503 contain supplementary X-ray crystallographic data for **22a** and **24** respectively. This data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, Union Road, Cambridge, CB2 1EZ; fax (+44) 1223-336-033 or email: deposit@ccdc.cam.ac.uk.

Suitable crystals for (**22h**) [both isomers], (**22i**) and (**23b**) were selected and data collected at 150(2) K on a Bruker Apex Duo diffractometer equipped with a graphite monochromated Mo(K*a*) radiation source and a cold stream of N_2 gas. Solutions were generated by conventional heavy atom Patterson or direct methods and refined by full-matrix least squares on all F^2 data, using SHELXS-97 and SHELXL software respectively.[v] Absorption corrections were applied based on multiple and symmetry-equivalent measurements using SADABS.[vi] CCDC1426769, CCDC1426768, CCDC1426767 and CCDC1420006 contain supplementary X-ray crystallographic data for **22h-D1**, **22h-D2**, **22i** and **23b** respectively. This data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, Union Road, Cambridge, CB2 1EZ; fax (+44) 1223-336-033 or email: deposit@ccdc.cam.ac.uk.

[i] Collect: Data collection software, R. Hooft, Nonius B.V., 1998
[ii] Z. Otwinowski & W. Minor, *Methods in Enzymology* (1997) Vol. **276**: *Macromolecular Crystallography*, part A, pp. 307–326; C. W. Carter, Jr. & R. M. Sweet, Eds., Academic Press
[iii] G. M. Sheldrick, *Acta Cryst.* (1990), ***A46***, 467–473
[iv] Sheldrick, G.M., *Acta Cryst*, (2015), ***C71***, 3-8
[v] SHELXTL Program System, Version 5.1, Bruker Analytical X-ray Instruments Inc., Madison, WI, 1998.
[vi] G. M. Sheldrick, SADABS: A Program for Absorption Correction with the Siemens SMART System, University of Göttingen (Germany), 1996.