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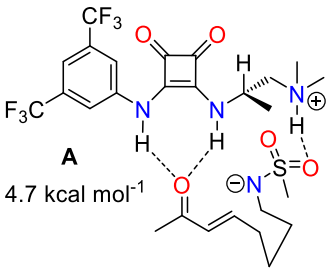
End Of Appendix 53

Energy calculations for transition states

XYZ Co-ordinates for Transition States **A-D** (calculated using B3LYP-D3(BJ)/def2-TZVPP//B3LYP/6-31G* as implemented in Gaussian 09).[1]

A

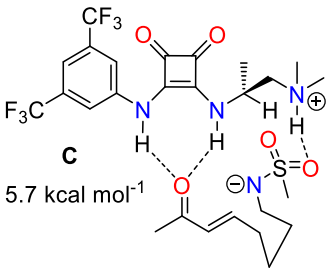


N	1.54109300	-3.34588000	-0.57461400
C	2.37270900	-3.90422400	0.53990100
H	1.71264900	-4.43489200	1.22764000
H	2.83266800	-3.07298400	1.07806300
C	0.73876500	-4.42011800	-1.23635600
H	0.13044000	-4.00953600	-2.04391200
H	0.09243400	-4.87042200	-0.48098900
C	2.38909400	-2.54520100	-1.52267100
C	1.61086900	-1.76088900	-2.59990200
H	3.09042400	-3.23677700	-2.00173000
N	0.54697600	-0.93696900	-2.03032800
C	-0.73713500	-1.08996200	-2.39432300
C	-1.92189600	-0.36897900	-2.15483800
C	-1.43946400	-2.11977500	-3.17661600
C	-2.73454500	-1.33929400	-2.95493700
O	-3.89923200	-1.46664700	-3.27678400
O	-1.09454800	-3.16596200	-3.71443700
N	-2.10272300	0.76749000	-1.44855800
H	-1.24717800	1.17350100	-1.04188100
H	0.89376900	-2.65366900	-0.08309900
H	0.77180700	-0.15071200	-1.40764400
O	0.44564000	1.50501300	-0.44223000
C	2.78298100	0.61595100	0.95820100
C	4.24187000	0.34409400	1.26564800

C	2.58274900	-0.46880400	3.70726600
N	2.09581100	-0.81788800	2.36412000
C	4.11201700	-0.41775200	3.70427400
C	4.68094300	0.60590400	2.71588700
H	4.48161900	-0.69531300	1.00939600
H	4.83159200	0.98098600	0.58965200
H	4.38390300	1.61915100	3.01851300
H	5.77728900	0.58262800	2.75914900
H	4.45878700	-0.18163800	4.71880400
H	4.49664000	-1.41725200	3.45884800
H	2.19758500	0.51598000	4.02909800
H	2.24492500	-1.20708600	4.44666900
S	0.56954800	-1.33021700	2.31528500
O	0.22736500	-1.55342100	0.86736600
O	0.32070200	-2.47607000	3.21635300
C	-0.56496800	-0.01613700	2.82146500
H	-1.57948700	-0.41382500	2.75397200
H	-0.43370600	0.82862700	2.14488400
H	-0.34320000	0.26908100	3.85208600
C	2.23303000	1.88936700	1.07164200
H	2.70879300	2.63556500	1.70117700
C	1.08487600	2.27758100	0.33053200
H	2.34475000	-0.00193000	0.18411200
C	0.60183300	3.71501000	0.45532600
H	1.33927700	4.36590200	0.93330400
H	-0.31950400	3.76060100	1.05060200
H	0.36305400	4.10830600	-0.53822500
H	2.96398000	-1.85676300	-0.89733800
H	1.42286400	-5.17473500	-1.63448900
H	3.12409500	-4.58126300	0.12658000
C	2.60585900	-0.90669700	-3.40206100
H	3.40286900	-1.52348900	-3.83316300
H	3.06748600	-0.14102300	-2.76777700

H	2.08360700	-0.40135800	-4.21871700
H	1.12583400	-2.46575400	-3.27972900
C	-3.28780200	1.45261400	-1.15397600
C	-5.55920400	2.95406600	-0.44448800
C	-3.16883400	2.58729400	-0.33617800
C	-4.55423000	1.06821900	-1.61901200
C	-5.66903700	1.82702700	-1.25904600
C	-4.29441900	3.32379100	0.01492800
H	-2.18994100	2.88709800	0.01914500
H	-4.66925800	0.18985300	-2.24827000
H	-6.43655700	3.52819300	-0.17337000
C	-7.01771400	1.45218500	-1.82185700
C	-4.15351000	4.48736600	0.95929100
F	-8.02667100	1.87459100	-1.02544000
F	-7.21623000	2.01288500	-3.03713400
F	-4.35920000	4.11340400	2.24396900
F	-2.91579700	5.03557100	0.90714700
F	-5.04220000	5.46635700	0.68868300
F	-7.14967800	0.12005600	-1.97285700

C

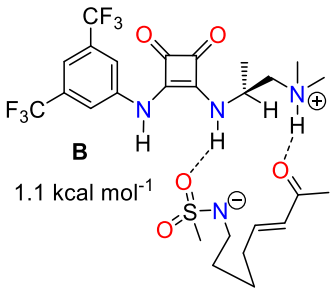


N	-1.94017600	3.18170900	0.63119100
C	-2.76489400	3.15385800	1.87673300
H	-2.09667900	3.29064800	2.72901600
H	-3.26305100	2.18585600	1.94401500
C	-1.16609800	4.45767400	0.52786900
H	-0.56227200	4.43011100	-0.38013500
H	-0.50231700	4.52790500	1.39168900
C	-2.74564400	2.88654900	-0.61244900
C	-1.97880000	2.02627400	-1.63739400
H	-3.02657300	3.84663200	-1.05549000
N	-1.65588100	0.69599100	-1.11744300
H	-0.66678700	0.42021000	-1.06619800
O	0.85938100	-0.74149500	-1.01391100
C	2.10982000	-0.68723000	-1.20588400
C	2.85972100	0.51251800	-1.31545300
H	3.92681700	0.42329700	-1.49775300
C	2.26638200	1.77537900	-1.27839800
C	2.89543000	2.96999700	-1.97015300
C	3.58323100	3.11544400	1.00723200
N	2.24108900	2.69633800	0.58074200
H	1.18271200	1.77194500	-1.26552700
H	2.98687900	2.70669800	-3.03398700
H	2.20880700	3.82303100	-1.91185100
H	-1.19385500	2.42996300	0.75802800
S	1.30613100	2.08841000	1.72711200
O	1.01230200	3.04683800	2.82293700
O	0.08643800	1.52707200	1.04977200

C	2.08223700	0.64710100	2.49673100
H	3.00471900	0.95804900	2.99165900
H	2.28983400	-0.08373600	1.71307900
H	1.38184000	0.24296900	3.23008100
C	-2.56777900	-0.22156200	-0.75071500
C	-2.45531300	-1.57001500	-0.35032300
C	-4.03119300	-0.21960400	-0.54539100
C	-3.92961200	-1.68664700	-0.13825800
O	-4.71258600	-2.54646500	0.21651300
O	-4.91829100	0.62090700	-0.63047500
C	4.26972300	3.40670200	-1.43588900
H	4.70820700	4.12487500	-2.14027800
H	4.95241900	2.54665400	-1.40933900
C	4.18663000	4.04732900	-0.04601700
H	5.18901300	4.34962500	0.28430900
H	3.57658900	4.95912200	-0.09965400
H	3.54001600	3.63883400	1.97250900
H	4.25062400	2.24439700	1.13533400
N	-1.35078800	-2.33899300	-0.24243700
H	-0.46287400	-1.88581000	-0.51260200
C	2.85826000	-2.00484100	-1.35780100
H	2.92832600	-2.51843500	-0.39030500
H	3.87443400	-1.87094100	-1.73889600
H	2.30678100	-2.66559300	-2.03497500
H	-3.50942400	3.95253700	1.83874700
H	-1.86375600	5.29797100	0.49615600
H	-3.66052700	2.36195500	-0.32602200
C	-2.78389700	1.96905300	-2.94661800
H	-3.78348200	1.56317600	-2.76402400
H	-2.26952000	1.33073100	-3.67034700
H	-2.89007600	2.96922700	-3.38207600
H	-1.00860600	2.49262800	-1.84378600
C	-1.23233400	-3.65963800	0.21124100

C	-0.79615800	-6.29832500	1.08450500
C	0.06938100	-4.17719100	0.30156100
C	-2.32204300	-4.46974200	0.56385200
C	-2.08637100	-5.77597100	0.99524300
C	0.27970000	-5.48172900	0.73390500
H	0.91035700	-3.55284700	0.02521900
H	-3.33792100	-4.09016000	0.49725200
H	-0.63404600	-7.31950700	1.40681700
C	-3.25517400	-6.62523000	1.42940400
C	1.68598800	-5.99736100	0.88389900
F	-3.54558500	-6.43088300	2.73681000
F	-2.99572200	-7.94458400	1.27820500
F	-4.37215300	-6.34780700	0.72916100
F	2.14223500	-5.83969800	2.14863900
F	2.55446900	-5.34809600	0.07285900
F	1.77133400	-7.31405700	0.59822600

B

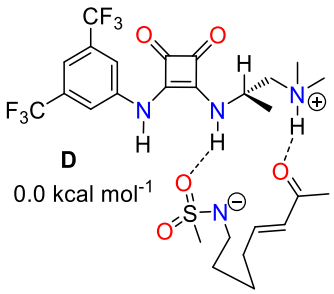


N	-2.36296000	3.10353700	-0.64035000
C	-1.79954100	4.20172500	-1.48434600
H	-1.68307800	3.83058000	-2.50297200
H	-0.82173300	4.47110000	-1.08635600
C	-3.63878100	2.57872800	-1.21278700
H	-3.93402600	1.68383600	-0.66841100
H	-3.45997400	2.30745600	-2.25462200
C	-2.50729100	3.56890700	0.78677700
C	-2.62795700	2.43911500	1.82309500
H	-3.39230800	4.21338900	0.82109000
N	-1.45280500	1.57149100	1.82186800
C	-0.18676500	1.96417800	2.03889900
C	1.02720500	1.25606800	2.03917400
C	0.49096500	3.24860600	2.31116300
C	1.81421800	2.48005900	2.35742500
O	2.98341000	2.75775000	2.54436700
O	0.13105700	4.41512500	2.39405500
N	1.23461400	-0.05507700	1.78008600
H	0.39928900	-0.58245800	1.50679200
H	-1.63115700	2.32917500	-0.70947000
H	-1.60346900	0.59801800	1.56453700
H	-4.40252900	3.35885200	-1.15745500
H	-2.47619500	5.05971900	-1.46446000
H	-1.62992900	4.17385300	1.03077500
O	-0.21316600	1.71598500	-1.11794800
C	-1.09960800	-0.61379100	-2.70133300

C	-1.43560200	-1.53627700	-3.86242500
C	-0.66323500	-3.04386600	-1.36472600
N	-1.65633700	-1.96529300	-1.33038900
C	-0.79431400	-3.75603500	-2.71717200
C	-0.58536400	-2.81976600	-3.91884000
H	-2.49707600	-1.80134900	-3.81736200
H	-1.28233600	-0.96589000	-4.79072900
H	0.47667100	-2.55481800	-3.99519600
H	-0.82725600	-3.36193500	-4.84186500
H	-0.07024000	-4.57986400	-2.76396400
H	-1.79666300	-4.20148300	-2.77098700
H	0.35213300	-2.63615300	-1.24222300
H	-0.81035100	-3.76914000	-0.55225600
S	-2.24768100	-1.50054400	0.07086200
O	-1.25770600	-1.37736100	1.20088600
O	-3.08403900	-0.28868300	-0.12829000
C	-3.37309700	-2.80726900	0.62987600
H	-3.78861400	-2.49933200	1.59191100
H	-2.81659700	-3.73942600	0.74760500
H	-4.16054400	-2.91991900	-0.11668800
C	0.21643500	-0.15506800	-2.53924500
H	1.02331100	-0.68989900	-3.02944700
C	0.57598300	0.98825000	-1.79252200
H	-1.89007200	0.06703800	-2.40186500
C	2.03724800	1.41416600	-1.79437600
H	2.65635600	0.81687900	-2.46917900
H	2.44836500	1.33432400	-0.78130400
H	2.10906100	2.46953500	-2.08092900
C	2.42630900	-0.78713300	1.74369800
C	4.71796800	-2.40932000	1.59249900
C	3.68610900	-0.24546700	2.03869500
C	2.31963000	-2.14075600	1.38574300
C	3.45748500	-2.93617800	1.30710600

C	4.81230000	-1.06615700	1.95853000
H	3.78495000	0.79900100	2.32296800
H	1.34340600	-2.55964400	1.16491400
H	5.60380500	-3.02899400	1.53125700
C	6.17095800	-0.46120700	2.20984600
C	3.30680300	-4.39414000	0.96464900
F	7.06785100	-1.39225700	2.60880300
F	6.12940400	0.49523100	3.15805900
F	4.41380700	-4.89183000	0.37370000
F	3.07401500	-5.14708900	2.06448500
F	2.26674700	-4.60674300	0.12151700
F	6.67129500	0.11081000	1.09000900
H	-3.45807600	1.77874100	1.55810900
C	-2.90071300	3.05909700	3.20419900
H	-2.95672400	2.26911000	3.95841400
H	-3.85182400	3.60401800	3.20521400
H	-2.10143100	3.75416100	3.47883100

D



N	-2.84121700	2.71417100	0.49607500
C	-2.51843300	3.76437000	1.51291900
H	-2.49021300	3.27907000	2.48785000
H	-1.53857100	4.19775300	1.30864000
C	-4.14787000	2.05634100	0.80100900
H	-4.29228700	1.24563600	0.08704100
H	-4.09114500	1.65210200	1.81228600
C	-2.81890800	3.22015300	-0.91759800
C	-1.43544200	3.68540200	-1.40167600
H	-3.16143200	2.38954200	-1.53708500
N	-0.45138000	2.61728500	-1.24739300
H	-1.08215300	4.52565900	-0.79900900
C	-1.53808000	4.13638600	-2.86643000
H	-0.56731500	4.50347500	-3.21041100
H	-1.83914600	3.30462600	-3.51404100
H	-2.27094800	4.94319600	-2.98112100
C	0.63173200	2.73316200	-0.46558700
C	1.71434500	1.89060500	-0.17190200
C	1.11550000	3.78872100	0.44172700
C	2.32480000	2.89750900	0.74580700
O	3.31550500	2.96729500	1.44727600
O	0.66757400	4.86471000	0.81295900
N	1.94382500	0.62882800	-0.60631100
H	1.18097400	0.21738200	-1.15270000
H	-2.12197400	1.94693200	0.64235900
H	-0.60137900	1.72991200	-1.73341300

H	-4.95166300	2.79362500	0.72992600
H	-3.29289600	4.53570900	1.48329900
H	-3.54014500	4.04244800	-0.97939100
O	-1.21530900	1.07028800	1.67229900
C	-2.08266600	-1.69512800	1.16402200
C	-2.66560500	-3.08513000	1.37513100
C	-0.58319500	-2.99766100	-0.79326200
N	-1.66863000	-2.01311300	-0.73044600
C	-1.11986500	-4.33238700	-0.26230700
C	-1.65745200	-4.23249200	1.17415400
H	-3.51840900	-3.22492000	0.70200300
H	-3.05424300	-3.12496800	2.40314500
H	-0.81825300	-4.11073800	1.87045100
H	-2.14024700	-5.17999700	1.44506000
H	-0.31905800	-5.08176500	-0.30772400
H	-1.92175500	-4.67131400	-0.93185000
H	0.26738100	-2.66154300	-0.18172400
H	-0.20575900	-3.13202500	-1.81621700
S	-1.71713500	-0.83082500	-1.79116600
O	-0.39289100	-0.18922900	-2.13116000
O	-2.77967900	0.13168400	-1.40159600
C	-2.24916800	-1.55823700	-3.36412700
H	-2.28392300	-0.76080100	-4.10982700
H	-1.53079100	-2.32157600	-3.67040800
H	-3.23714000	-1.99662700	-3.21632200
C	-0.97362600	-1.28930100	1.92916700
H	-0.35947500	-2.04506100	2.40798600
C	-0.64082500	0.05845200	2.17808300
H	-2.80780500	-0.92164500	0.93180500
C	0.47493900	0.34887600	3.17276200
H	0.92373800	-0.55660100	3.59023300
H	1.26140400	0.94485800	2.69639800
H	0.07620800	0.95693600	3.99364500

C	3.00618400	-0.23685400	-0.32613000
C	5.03508700	-2.12436600	0.15696900
C	4.09783300	0.10279900	0.48662300
C	2.94695100	-1.51375100	-0.90635100
C	3.94768300	-2.44653700	-0.65580900
C	5.09558100	-0.84613900	0.71363600
H	4.16237600	1.08830600	0.94035700
H	2.10868500	-1.77162600	-1.54456500
H	5.81466500	-2.84971700	0.35251400
C	6.23050900	-0.49434800	1.64301200
C	3.86848300	-3.79605100	-1.31766700
F	7.32311800	-1.25805500	1.41200500
F	6.60193500	0.79503500	1.51642800
F	4.53797400	-4.74007900	-0.62407900
F	4.39023600	-3.77655600	-2.56559100
F	2.58481300	-4.21870600	-1.44207800
F	5.88546500	-0.68337200	2.93741200

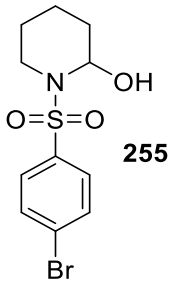
[1] Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, J. A., Jr.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, J. M.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, O¨.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. Gaussian 09, Gaussian, Inc., Wallingford, CT, **2009**.

General statement about X-ray structures

X-Ray crystallography was performed by Dr William Lewis.

A suitable crystal was selected and placed in fomblin film on a micromount on aDual Cu SuperNova Atlas diffractometer. The crystal was kept at 120(2) K during data collection. Using Olex2,[2] the structure was solved with the olex2.solve[3] structure solution program using Charge Flipping and refined with the ShelXL[4] refinement package using Least Squares minimisation.

X-Ray data for 1-((4-bromophenyl)sulfonyl)piperidin-2-ol 255



255

Table 1 Crystal data and structure refinement for 255.

Identification code	**255**
Empirical formula	$C_{11}H_{14}BrNO_3S$
Formula weight	320.20
Temperature/K	120(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	6.1298(4)
b/Å	12.3479(7)
c/Å	16.3172(9)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1235.05(13)
Z	4
ρ_{calc}g/cm^3	1.722
μ/mm^{-1}	6.106
F(000)	648.0
Crystal size/mm^3	0.9243 × 0.3089 × 0.088
Radiation	CuKα (λ = 1.54184)

2Θ range for data collection/°	8.982 to 147.94
Index ranges	-6 ≤ h ≤ 7, -13 ≤ k ≤ 15, -20 ≤ l ≤ 11
Reflections collected	4385
Independent reflections	2414 [R_{int} = 0.0420, R_{sigma} = 0.0444]
Data/restraints/parameters	2414/4/166
Goodness-of-fit on F^2	1.061
Final R indexes [I>=2σ (I)]	R_1 = 0.0407, wR_2 = 0.1075
Final R indexes [all data]	R_1 = 0.0410, wR_2 = 0.1080
Largest diff. peak/hole / e $Å^{-3}$	0.43/-0.76
Flack parameter	0.02(2)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters ($Å^2$×10^3) for 255. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*Y*	*z*	U(eq)
N1	8029(6)	6413(3)	6491(2)	15.4(8)
C2	5860(8)	6724(4)	6788(3)	24.8(10)
C3	6051(12)	7890(5)	7112(4)	35.2(14)
C4	6870(11)	8656(5)	6460(4)	39.1(15)
C5	9070(11)	8263(5)	6125(5)	42.2(16)
C6	8919(10)	7094(4)	5832(4)	30.1(11)
O7	4273(8)	6696(5)	6209(4)	29.7(15)
O7A	7880(20)	7084(13)	5178(8)	36(4)
S8	8670.7(18)	5131.5(9)	6494.1(6)	15.2(3)
O9	7783(6)	4673(3)	7227(2)	22.2(8)
O10	10979(6)	5064(3)	6343(2)	24.8(8)
C11	7354(8)	4528(4)	5652(3)	16.5(9)
C12	5291(8)	4080(4)	5748(3)	19.6(10)
C13	4201(8)	3656(4)	5078(3)	20.0(9)
C14	5194(8)	3712(4)	4314(3)	19.0(9)
C15	7248(9)	4154(4)	4205(3)	22.1(10)

C16	8330(8)	4556(4)	4879(3)	20.3(10)
Br17	3652.6(9)	3161.7(4)	3389.1(3)	26.8(2)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 255. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka*b*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	19.3(17)	13.0(17)	13.9(19)	1.6(16)	-1.0(14)	-0.5(14)
C2	28(2)	20(2)	27(2)	8(2)	8(2)	9(2)
C3	51(4)	25(3)	29(3)	-6(2)	0(3)	14(3)
C4	59(4)	15(2)	43(4)	0(3)	-18(3)	5(3)
C5	47(3)	19(3)	61(4)	12(3)	-6(3)	-13(3)
C6	32(3)	23(3)	35(3)	9(2)	7(2)	-2(2)
O7	24(2)	23(3)	42(3)	7(2)	-9(2)	-4(2)
O7A	49(8)	40(9)	21(6)	10(5)	6(5)	2(6)
S8	18.5(5)	13.6(5)	13.5(5)	1.9(4)	-1.2(5)	1.3(4)
O9	36.4(19)	17.9(16)	12.2(16)	6.7(14)	-1.2(14)	1.3(15)
O10	17.8(17)	27.2(18)	29.3(19)	-3.7(16)	-1.7(14)	4.3(14)
C11	21(2)	11(2)	17(2)	4.5(18)	1.8(17)	3.0(19)
C12	20(2)	21(2)	18(2)	1.3(19)	4.7(17)	1.4(19)
C13	20(2)	18(2)	22(2)	4.3(19)	5.2(18)	1.6(18)
C14	25(2)	13(2)	19(2)	-0.2(18)	0.1(18)	4.8(19)
C15	31(2)	20(2)	15(2)	-1(2)	6.2(19)	-2(2)
C16	24(2)	19(2)	18(2)	1.5(19)	6.0(18)	0(2)
Br17	38.4(3)	23.0(3)	19.0(3)	-5.07(19)	-2.5(2)	-6.1(2)

Table 4 Bond Lengths for 255.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C2	1.466(6)	S8	O10	1.439(4)
N1	C6	1.471(6)	S8	C11	1.760(5)
N1	S8	1.630(4)	C11	C12	1.389(7)

C2	C3	1.539(7)	C11	C16	1.396(6)
C2	O7	1.356(8)	C12	C13	1.383(7)
C3	C4	1.510(9)	C13	C14	1.390(7)
C4	C5	1.534(10)	C14	C15	1.384(7)
C5	C6	1.523(8)	C14	Br17	1.906(5)
C6	O7A	1.243(14)	C15	C16	1.378(7)
S8	O9	1.430(4)			

Table 5 Bond Angles for 255.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C2	N1	C6	115.3(4)	O9	S8	O10	119.6(2)
C2	N1	S8	118.2(3)	O9	S8	C11	108.1(2)
C6	N1	S8	117.9(3)	O10	S8	N1	107.0(2)
N1	C2	C3	106.9(5)	O10	S8	C11	107.0(2)
O7	C2	N1	114.4(5)	C12	C11	S8	119.9(4)
O7	C2	C3	108.6(5)	C12	C11	C16	120.1(5)
C4	C3	C2	111.6(5)	C16	C11	S8	119.9(4)
C3	C4	C5	110.2(5)	C13	C12	C11	120.1(4)
C6	C5	C4	111.0(5)	C12	C13	C14	118.6(5)
N1	C6	C5	109.6(5)	C13	C14	Br17	118.4(4)
O7A	C6	N1	115.6(9)	C15	C14	C13	122.3(5)
O7A	C6	C5	108.1(9)	C15	C14	Br17	119.3(4)
N1	S8	C11	107.3(2)	C16	C15	C14	118.5(4)
O9	S8	N1	107.1(2)	C15	C16	C11	120.4(5)

Table 6 Hydrogen Bonds for 255.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O7	H7	O10[1]	0.84	2.04	2.861(7)	167.0
O7A	H7A	O7[2]	0.84	2.04	2.849(15)	162.9

[1]-1+X,+Y,+Z; [2]1/2+X,3/2-Y,1-Z

Table 7 Torsion Angles for 255.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
N1	C2	C3	C4	57.6(6)	S8	N1	C2	C3	153.0(4)
N1	S8	C11	C12	-91.3(4)	S8	N1	C2	O7	-86.8(5)
N1	S8	C11	C16	85.0(4)	S8	N1	C6	C5	-153.8(4)
C2	N1	C6	C5	58.8(6)	S8	N1	C6	O7A	83.8(10)
C2	N1	C6	O7A	-63.6(11)	S8	C11	C12	C13	176.5(4)
C2	N1	S8	O9	-38.8(4)	S8	C11	C16	C15	-175.4(4)
C2	N1	S8	O10	-168.3(4)	O9	S8	C11	C12	24.0(4)
C2	N1	S8	C11	77.1(4)	O9	S8	C11	C16	-159.7(4)
C2	C3	C4	C5	-56.7(7)	O10	S8	C11	C12	154.1(4)
C3	C4	C5	C6	53.9(7)	O10	S8	C11	C16	-29.6(5)
C4	C5	C6	N1	-53.3(7)	C11	C12	C13	C14	-1.4(7)
C4	C5	C6	O7A	73.5(10)	C12	C11	C16	C15	0.9(7)
C6	N1	C2	C3	-59.7(6)	C12	C13	C14	C15	1.4(7)
C6	N1	C2	O7	60.5(6)	C12	C13	C14	Br17	-178.4(4)
C6	N1	S8	O9	174.8(4)	C13	C14	C15	C16	-0.3(7)
C6	N1	S8	O10	45.3(4)	C14	C15	C16	C11	-0.9(7)
C6	N1	S8	C11	-69.3(4)	C16	C11	C12	C13	0.2(7)
O7	C2	C3	C4	-66.3(7)	Br17	C14	C15	C16	179.5(4)

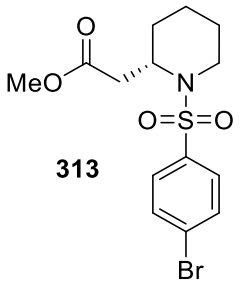
Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 255.

Atom	*x*	*y*	*z*	U(eq)
H2BA	5382	6231	7231	30
H2BB	4782	6689	6337	30
H2A	5438	6236	7250	30
H3A	4604	8138	7307	42
H3B	7064	7903	7585	42
H4A	7039	9391	6694	47
H4B	5796	8697	6008	47
H5A	9525	8732	5663	51
H5B	10191	8318	6560	51
H6AA	7963	7052	5344	36
H6AB	10386	6830	5675	36
H6B	10434	6837	5713	36
H7	3453	6163	6295	45
H7A	8507	7485	4834	54
H12	4627	4064	6274	24
H13	2803	3334	5140	24
H15	7897	4180	3676	26
H16	9750	4854	4818	24

Table 9 Atomic Occupancy for 255.

Atom	*Occupancy*	Atom	*Occupancy*	Atom	*Occupancy*
H2BA	0.291(10)	H2BB	0.291(10)	H2A	0.709(10)
H6AA	0.709(10)	H6AB	0.709(10)	H6B	0.291(10)
O7	0.709(10)	H7	0.709(10)	O7A	0.291(10)
H7A	0.291(10)				

X-Ray data for methyl (*S*)-2-(1-((4-bromophenyl)sulfonyl)piperidin-2-yl)acetate **313**



313

Table 1 Crystal data and structure refinement for 313.

Identification code	313
Empirical formula	$C_{14}H_{18}NO_4SBr$
Formula weight	376.26
Temperature/K	120(2)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	7.80772(8)
b/Å	20.2961(2)
c/Å	10.05075(9)
α/°	90
β/°	94.5595(9)
γ/°	90
Volume/Å^3	1587.66(3)
Z	4
ρ_{calc}g/cm^3	1.574
μ/mm^{-1}	4.895
F(000)	768.0
Crystal size/mm^3	0.212 × 0.185 × 0.074
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.714 to 153.932
Index ranges	-9 ≤ h ≤ 9, -25 ≤ k ≤ 24, -12 ≤ l ≤ 12
Reflections collected	19225
Independent reflections	6327 [R_{int} = 0.0278, R_{sigma} = 0.0233]
Data/restraints/parameters	6327/319/381
Goodness-of-fit on F^2	1.067
Final R indexes [I>=2σ (I)]	R_1 = 0.0325, wR_2 = 0.0888
Final R indexes [all data]	R_1 = 0.0331, wR_2 = 0.0895
Largest diff. peak/hole / e Å^{-3}	0.25/-0.59
Flack parameter	-0.035(10)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 313. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*X*	*Y*	*z*	U(eq)
N1	2340(4)	5000.3(16)	2837(3)	38.1(6)
C2	3989(5)	4646(2)	2770(4)	41.7(8)
C3	4808(6)	4801(3)	1470(5)	54.2(11)
C4	4969(7)	5532(3)	1241(5)	62.4(13)
C5	3242(8)	5857(3)	1254(6)	68.2(14)
C6	2386(7)	5715(2)	2537(6)	59.0(11)
S7	537.3(11)	4609.1(5)	2522.4(9)	37.14(19)
O8	749(4)	3965.1(16)	3078(3)	48.3(7)
O9	-794(4)	5020.7(19)	2938(3)	56.1(8)
C10	172(4)	4517.3(18)	772(4)	35.2(7)
C11	790(5)	3964(2)	157(4)	39.2(8)
C12	571(6)	3903(2)	-1209(4)	47.0(9)
C13	-243(5)	4408(2)	-1947(4)	46.5(9)
C14	-876(6)	4957(2)	-1352(5)	51.6(10)
C15	-675(5)	5009(2)	25(4)	45.5(9)
Br16	-444.1(8)	4341.9(4)	-3838.8(5)	78.9(2)
C17	5191(6)	4828(3)	4006(5)	54.3(11)
C18	4687(5)	4509(2)	5264(4)	46.3(9)
O19	4235(6)	3952.4(19)	5354(4)	72.9(11)
O20	4879(5)	4919.4(17)	6296(3)	57.9(8)
C21	4550(10)	4656(4)	7578(6)	79.5(17)
N31	4670(4)	7089.8(18)	8019(3)	42.6(7)
C32	3871(7)	7746(3)	7752(6)	63.3(13)
C33	3010(8)	7770(5)	6319(7)	99(2)
C34	1861(9)	7178(6)	5998(8)	117(3)
C35	2801(9)	6537(6)	6229(8)	111(3)
C36	3579(8)	6512(3)	7641(7)	74.6(16)
S37	6721.4(11)	7000.3(6)	8007.9(8)	37.97(18)
O38	7513(4)	7566.5(18)	8618(3)	56.7(8)
O39	7135(5)	6362.6(18)	8548(3)	59.6(9)
C40	7249(4)	6992(2)	6324(3)	35.0(6)
C41	7404(5)	7585(2)	5671(5)	40.5(8)
C42	7731(6)	7587(2)	4331(5)	43.2(9)
C43	7938(5)	6995(2)	3696(4)	40.3(7)
C44	7835(6)	6396(2)	4357(4)	41.7(9)
C45	7464(6)	6395.3(19)	5671(4)	39.7(8)
Br46	8366.1(8)	6995.7(3)	1863.1(5)	63.08(16)
C47	2602(9)	7881(4)	8807(7)	86.3(19)
C48	3443(9)	7999(3)	10159(7)	75.6(16)
O49	4615(11)	8364(3)	10412(7)	132(3)
O50	2734(6)	7647(2)	11078(5)	78.1(11)
C51	3422(13)	7747(5)	12429(8)	107(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 313. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	45.9(16)	35.2(16)	33.9(15)	-3.2(12)	6.2(12)	-3.7(12)
C2	40.2(17)	43(2)	41.6(19)	-1.0(16)	3.2(14)	-4.0(15)
C3	48(2)	70(3)	46(2)	-9(2)	10.5(17)	-10(2)
C4	70(3)	77(3)	41(2)	8(2)	6(2)	-30(2)
C5	87(4)	53(3)	63(3)	16(2)	1(3)	-20(2)
C6	78(3)	35(2)	64(3)	-3.4(19)	6(2)	-6.4(19)
S7	38.4(4)	43.6(5)	30.3(4)	-1.0(3)	8.4(3)	-2.3(3)
O8	55.3(16)	49.9(16)	39.3(14)	11.4(12)	1.8(12)	-14.5(13)
O9	47.5(15)	77(2)	46.1(17)	-13.1(15)	15.7(13)	8.0(15)
C10	36.4(16)	36.3(18)	33.5(16)	1.0(13)	5.8(12)	-0.5(13)
C11	48(2)	32.1(18)	37.7(19)	2.2(14)	1.4(15)	4.5(15)
C12	56(2)	45(2)	41(2)	-6.7(16)	6.6(17)	6.9(17)
C13	45.2(19)	61(3)	33.8(18)	-1.1(18)	4.1(15)	4.3(18)
C14	58(2)	56(3)	41(2)	5.8(18)	0.3(18)	15(2)
C15	48(2)	44(2)	44(2)	-2.1(16)	4.1(16)	13.1(17)
Br16	79.8(4)	124.3(6)	31.5(2)	-3.1(3)	-1.6(2)	13.3(4)
C17	48(2)	65(3)	48(2)	13(2)	-4.4(17)	-22(2)
C18	46(2)	45(2)	47(2)	9.8(16)	-4.4(16)	-11.5(16)
O19	109(3)	47.6(19)	60(2)	7.4(15)	-9(2)	-29(2)
O20	77(2)	43.5(17)	53.5(19)	3.5(13)	7.8(15)	-9.7(15)
C21	104(5)	84(4)	52(3)	6(3)	17(3)	-16(3)
N31	42.4(15)	46(2)	39.9(17)	0.8(13)	6.0(12)	3.2(13)
C32	61(3)	69(3)	62(3)	23(2)	19(2)	27(2)
C33	59(3)	170(7)	71(4)	48(4)	13(3)	38(4)
C34	54(3)	236(9)	61(4)	18(5)	1(3)	-11(4)
C35	62(4)	191(8)	79(4)	-31(5)	-1(3)	-33(4)
C36	67(3)	76(4)	81(4)	-15(3)	8(3)	-22(3)
S37	42.6(4)	38.6(4)	32.1(4)	-1.7(4)	-1.0(3)	4.7(4)
O38	59.6(18)	62(2)	47.8(18)	-17.4(15)	-2.6(14)	-10.9(15)
O39	81(2)	55(2)	43.8(17)	15.5(14)	7.5(15)	26.1(16)
C40	34.8(15)	32.9(15)	37.0(16)	-1.0(16)	1.2(12)	-0.9(15)
C41	44(2)	30.3(19)	48(2)	-2.2(15)	4.9(16)	-0.2(14)
C42	49(2)	34(2)	47(2)	3.9(16)	9.8(17)	-1.2(16)
C43	40.3(17)	42.2(18)	38.9(18)	-0.8(18)	6.9(13)	-1.2(17)
C44	50(2)	33(2)	42(2)	-3.9(15)	7.8(17)	-2.1(15)
C45	51(2)	28.3(18)	40(2)	0.1(14)	6.4(16)	0.0(15)
Br46	90.0(4)	59.2(3)	42.9(2)	2.5(2)	23.1(2)	-2.5(3)
C47	80(4)	98(5)	85(4)	9(3)	30(3)	43(3)
C48	100(4)	49(3)	83(4)	-13(2)	37(3)	13(3)
O49	188(6)	105(4)	111(5)	-46(4)	67(4)	-66(4)
O50	80(3)	78(3)	77(3)	11(2)	10(2)	1(2)
C51	127(7)	115(6)	80(5)	10(4)	6(4)	-6(5)

Table 4 Bond Lengths for 313.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C2	1.481(5)	N31	C32	1.487(6)
N1	C6	1.483(5)	N31	C36	1.482(7)
N1	S7	1.625(3)	N31	S37	1.613(3)
C2	C3	1.532(6)	C32	C33	1.541(9)
C2	C17	1.542(6)	C32	C47	1.532(8)
C3	C4	1.508(8)	C33	C34	1.518(14)
C4	C5	1.503(9)	C34	C35	1.501(16)
C5	C6	1.527(8)	C35	C36	1.500(11)
S7	O8	1.426(3)	S37	O38	1.421(3)
S7	O9	1.422(3)	S37	O39	1.431(3)
S7	C10	1.769(4)	S37	C40	1.773(4)
C10	C11	1.386(5)	C40	C41	1.382(6)
C10	C15	1.386(6)	C40	C45	1.393(6)
C11	C12	1.376(6)	C41	C42	1.391(7)
C12	C13	1.389(6)	C42	C43	1.376(7)
C13	C14	1.374(7)	C43	C44	1.392(6)
C13	Br16	1.901(4)	C43	Br46	1.898(4)
C14	C15	1.384(6)	C44	C45	1.374(7)
C17	C18	1.501(6)	C47	C48	1.481(11)
C18	O19	1.190(5)	C48	O49	1.189(9)
C18	O20	1.329(6)	C48	O50	1.323(8)
O20	C21	1.437(7)	O50	C51	1.434(10)

Table 5 Bond Angles for 313.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C2	N1	C6	115.5(3)	C32	N31	S37	120.1(3)
C2	N1	S7	119.7(3)	C36	N31	C32	116.1(4)
C6	N1	S7	118.2(3)	C36	N31	S37	117.4(4)
N1	C2	C3	111.3(4)	N31	C32	C33	110.0(5)
N1	C2	C17	108.8(3)	N31	C32	C47	108.7(4)
C3	C2	C17	111.6(3)	C47	C32	C33	112.5(5)
C4	C3	C2	112.2(4)	C34	C33	C32	112.4(6)
C5	C4	C3	110.2(4)	C35	C34	C33	112.3(6)
C4	C5	C6	112.2(4)	C36	C35	C34	109.4(8)
N1	C6	C5	112.1(4)	N31	C36	C35	113.4(6)
N1	S7	C10	108.28(16)	N31	S37	C40	108.30(17)
O8	S7	N1	107.44(17)	O38	S37	N31	107.7(2)
O8	S7	C10	107.32(18)	O38	S37	O39	119.4(2)
O9	S7	N1	107.32(19)	O38	S37	C40	107.1(2)
O9	S7	O8	119.2(2)	O39	S37	N31	107.1(2)
O9	S7	C10	106.86(18)	O39	S37	C40	106.9(2)
C11	C10	S7	119.6(3)	C41	C40	S37	118.7(3)
C15	C10	S7	119.7(3)	C41	C40	C45	121.0(3)
C15	C10	C11	120.7(4)	C45	C40	S37	120.3(3)
C12	C11	C10	119.8(4)	C40	C41	C42	119.6(3)
C11	C12	C13	118.8(4)	C43	C42	C41	118.9(4)

C12	C13	Br16	118.6(3)	C42	C43	C44	121.9(3)
C14	C13	C12	122.1(4)	C42	C43	Br46	119.1(3)
C14	C13	Br16	119.3(3)	C44	C43	Br46	119.0(3)
C13	C14	C15	118.8(4)	C45	C44	C43	119.0(4)
C14	C15	C10	119.9(4)	C44	C45	C40	119.7(4)
C18	C17	C2	113.1(3)	C48	C47	C32	113.6(6)
O19	C18	C17	124.9(5)	O49	C48	C47	125.0(7)
O19	C18	O20	123.5(4)	O49	C48	O50	123.0(7)
O20	C18	C17	111.5(4)	O50	C48	C47	112.0(6)
C18	O20	C21	116.7(4)	C48	O50	C51	116.0(6)

Table 6 Torsion Angles for 313.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
N1	C2	C3	C4	52.5(5)	N31	C32	C33	C34	49.1(7)
N1	C2	C17	C18	73.8(5)	N31	C32	C47	C48	69.9(8)
N1	S7	C10	C11	90.1(3)	N31	S37	C40	C41	81.6(3)
N1	S7	C10	C15	-87.7(3)	N31	S37	C40	C45	-97.0(3)
C2	N1	C6	C5	48.9(6)	C32	N31	C36	C35	51.9(7)
C2	N1	S7	O8	38.3(3)	C32	N31	S37	O38	39.3(4)
C2	N1	S7	O9	167.6(3)	C32	N31	S37	O39	168.8(4)
C2	N1	S7	C10	-77.3(3)	C32	N31	S37	C40	-76.2(4)
C2	C3	C4	C5	-56.2(5)	C32	C33	C34	C35	-55.3(8)
C2	C17	C18	O19	42.4(7)	C32	C47	C48	O49	48.3(10)
C2	C17	C18	O20	-140.4(4)	C32	C47	C48	O50	-131.7(6)
C3	C2	C17	C18	-163.0(4)	C33	C32	C47	C48	-168.0(6)
C3	C4	C5	C6	55.5(6)	C33	C34	C35	C36	56.1(8)
C4	C5	C6	N1	-51.5(6)	C34	C35	C36	N31	-53.6(8)
C6	N1	C2	C3	-49.2(5)	C36	N31	C32	C33	-48.0(6)
C6	N1	C2	C17	74.2(4)	C36	N31	C32	C47	75.6(6)
C6	N1	S7	O8	-171.3(3)	C36	N31	S37	O38	-170.0(4)
C6	N1	S7	O9	-41.9(4)	C36	N31	S37	O39	-40.4(4)
C6	N1	S7	C10	73.1(3)	C36	N31	S37	C40	74.5(4)
S7	N1	C2	C3	102.0(4)	S37	N31	C32	C33	103.1(5)
S7	N1	C2	C17	-134.6(3)	S37	N31	C32	C47	-133.3(5)
S7	N1	C6	C5	-102.8(5)	S37	N31	C36	C35	-100.1(6)
S7	C10	C11	C12	-177.3(3)	S37	C40	C41	C42	-176.8(3)
S7	C10	C15	C14	176.3(3)	S37	C40	C45	C44	178.6(3)
O8	S7	C10	C11	-25.6(3)	O38	S37	C40	C41	-34.3(4)
O8	S7	C10	C15	156.6(3)	O38	S37	C40	C45	147.1(3)
O9	S7	C10	C11	-154.6(3)	O39	S37	C40	C41	-163.3(3)
O9	S7	C10	C15	27.6(4)	O39	S37	C40	C45	18.1(4)
C10	C11	C12	C13	1.2(6)	C40	C41	C42	C43	-1.7(7)
C11	C10	C15	C14	-1.5(7)	C41	C40	C45	C44	0.0(6)
C11	C12	C13	C14	-1.8(7)	C41	C42	C43	C44	-0.2(6)
C11	C12	C13	Br16	177.0(3)	C41	C42	C43	Br46	179.5(3)
C12	C13	C14	C15	0.8(7)	C42	C43	C44	C45	2.0(6)
C13	C14	C15	C10	0.9(7)	C43	C44	C45	C40	-1.9(6)
C15	C10	C11	C12	0.4(6)	C45	C40	C41	C42	1.9(6)

Br16	C13	C14	C15	-178.0(4)	Br46	C43	C44	C45	-177.7(3)
C17	C2	C3	C4	-69.2(5)	C47	C32	C33	C34	-72.2(8)
C17	C18	O20	C21	-175.8(5)	C47	C48	O50	C51	-177.7(6)
O19	C18	O20	C21	1.5(7)	O49	C48	O50	C51	2.2(10)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 313.

Atom	*x*	*y*	*z*	U(eq)
H2	3755	4162	2796	50
H3A	5963	4598	1502	65
H3B	4101	4604	711	65
H4A	5770	5725	1948	75
H4B	5444	5611	370	75
H5A	2488	5698	481	82
H5B	3380	6339	1157	82
H6A	3022	5947	3289	71
H6B	1197	5889	2451	71
H11	1363	3628	678	47
H12	969	3523	-1641	56
H14	-1441	5294	-1875	62
H15	-1118	5382	456	55
H17A	6378	4694	3851	65
H17B	5185	5312	4122	65
H21A	4737	5001	8258	119
H21B	5330	4287	7797	119
H21C	3358	4502	7554	119
H32	4795	8089	7838	76
H33A	2316	8177	6205	119
H33B	3910	7786	5680	119
H34A	878	7192	6562	140
H34B	1399	7202	5054	140
H35A	1994	6165	6062	133
H35B	3715	6499	5605	133
H36A	2645	6483	8250	90
H36B	4282	6107	7761	90
H41	7288	7988	6134	49
H42	7809	7991	3861	52
H44	8018	5993	3908	50
H45	7355	5991	6131	48
H47A	1901	8272	8531	104
H47B	1816	7500	8843	104
H51A	4659	7832	12443	161
H51B	3224	7352	12957	161
H51C	2853	8126	12809	161

X-Ray data for (*S*)-1-(4-bromophenyl)-2-(1-((4-nitrophenyl)sulfonyl)piperidin-2-yl)ethan-1-one **283**

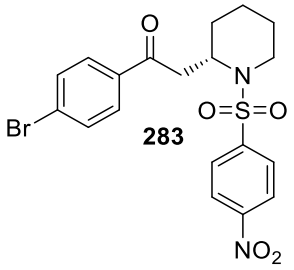


Table 1 Crystal data and structure refinement for 283.

Identification code	**283**
Empirical formula	$C_{19}H_{19}BrN_2O_5S$
Formula weight	467.33
Temperature/K	120(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	5.9640(2)
b/Å	13.5921(5)
c/Å	23.4651(11)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1902.16(14)
Z	4
ρ_{calc}g/cm^3	1.632
μ/mm^{-1}	4.281
F(000)	952.0
Crystal size/mm^3	0.4765 × 0.1066 × 0.015
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.516 to 148.766
Index ranges	-4 ≤ h ≤ 7, -16 ≤ k ≤ 16, -28 ≤ l ≤ 28
Reflections collected	7263
Independent reflections	3754 [R_{int} = 0.0277, R_{sigma} = 0.0346]
Data/restraints/parameters	3754/0/253
Goodness-of-fit on F^2	1.031
Final R indexes [I>=2σ (I)]	R_1 = 0.0350, wR_2 = 0.0901
Final R indexes [all data]	R_1 = 0.0370, wR_2 = 0.0921
Largest diff. peak/hole / e Å^{-3}	0.54/-0.35
Flack parameter	-0.014(12)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 283. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br1	-73.1(8)	6523.6(3)	5684.5(2)	38.28(15)
S2	2614.9(13)	3383.0(6)	2493.9(4)	21.60(19)
O3	5001(5)	3300.7(18)	2480.2(12)	28.6(5)
O4	5790(4)	5372(2)	3279.2(14)	29.5(6)
O5	1344(6)	3399(3)	5311.0(14)	46.5(8)

N6	-20(6)	3670(2)	4951.9(14)	32.1(7)
C	1830(6)	3473(3)	3224.9(16)	21.3(7)
C1	-443(6)	4554(3)	2005.8(17)	28.1(8)
O9	1282(5)	2609(2)	2256.6(14)	32.8(7)
O10	-1883(6)	3987(3)	5064.5(15)	42.8(8)
C2	3912(6)	5716(3)	3306.3(18)	22.0(7)
C3	2799(6)	3309(3)	4214.4(18)	28.2(8)
N13	1904(5)	4396(2)	2186.5(14)	20.7(6)
C4	639(6)	3599(3)	4348.5(16)	24.3(7)
C5	2909(6)	5962(3)	3871.1(18)	22.0(7)
C6	3392(6)	3252(3)	3643.7(18)	25.5(8)
C7	-336(6)	3756(3)	3368.9(17)	25.1(7)
C8	-75(7)	6573(3)	4472.2(18)	29.4(7)
C9	2571(7)	5918(3)	2770.5(18)	26.0(8)
C10	791(6)	6397(3)	3936.3(18)	27.4(8)
C11	-482(7)	5123(4)	1451.5(18)	34.4(9)
C12	-922(6)	3831(3)	3935.7(18)	25.3(8)
C13	4112(7)	5719(3)	4361.7(19)	28.3(8)
C14	3250(8)	5888(3)	4899(2)	31.4(9)
C15	1150(7)	6306(3)	4945.3(19)	29.2(8)
C16	3212(7)	5864(3)	1696(2)	29.8(9)
C17	806(7)	6083(3)	1508.2(19)	33.5(9)
C18	3300(6)	5297(3)	2255.5(17)	22.1(7)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 283. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka*b*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br1	49.9(3)	31.3(2)	33.6(2)	2.32(17)	10.7(2)	1.8(2)
S2	19.6(4)	16.6(4)	28.6(4)	-3.2(3)	4.4(3)	-1.4(3)
O3	22.6(12)	23.5(11)	39.9(14)	2.5(11)	7.7(12)	6.4(11)
O4	19.9(12)	33.1(14)	35.4(15)	-1.8(13)	-3.2(11)	5.7(11)
O5	46.1(18)	67(2)	26.7(15)	8.4(17)	-7.8(13)	-12.1(19)
N6	36.6(17)	32.2(16)	27.5(16)	2.1(13)	-3.1(16)	-10.3(17)
C	19.3(15)	15.1(15)	29.4(18)	-1.5(14)	1.3(13)	-2.7(14)
C1	16.6(17)	38(2)	29.8(19)	-3.3(16)	1.3(14)	-1.0(15)
O9	36.5(16)	23.4(13)	38.4(16)	-8.2(12)	6.1(13)	-11.7(12)
O10	47.0(19)	53(2)	28.7(16)	-0.2(15)	7.3(15)	4.3(16)
C2	18.8(17)	15.6(15)	32(2)	1.2(15)	-5.3(15)	-2.1(13)
C3	27.6(18)	24.3(18)	33(2)	5.1(15)	-7.6(15)	-1.5(15)
N13	14.7(13)	22.6(15)	24.8(16)	-2.9(13)	-0.1(11)	-4.0(11)
C4	29.0(17)	19.1(15)	24.8(17)	2.7(15)	-0.9(14)	-4.8(13)
C5	21.2(18)	15.7(16)	29.2(19)	-2.7(13)	-2.9(15)	-1.4(13)
C6	16.8(15)	19.6(17)	40(2)	2.0(15)	-2.5(15)	2.0(13)
C7	19.1(17)	27.0(17)	29.2(18)	-0.5(14)	-2.9(15)	2.4(14)
C8	24.4(16)	26.3(17)	37.6(19)	-4.6(15)	-0.9(15)	4(2)
C9	27.5(18)	20.3(17)	30.1(19)	-4.8(14)	-7.6(17)	8.5(15)
C10	22.4(16)	23.7(18)	36(2)	-1.6(16)	-7.8(15)	3.5(15)
C11	22.8(19)	54(3)	25.9(19)	-1.5(18)	-2.6(15)	6.8(18)
C12	21.4(16)	25.5(18)	29(2)	-0.7(15)	0.0(15)	0.9(15)

C13	28.0(18)	22.2(16)	35(2)	-2.1(16)	-7.4(17)	3.4(15)
C14	36(2)	24.3(19)	34(2)	3.8(16)	-7.5(19)	5.4(16)
C15	37(2)	21.0(17)	30(2)	-2.1(15)	4.1(17)	-2.9(16)
C16	31(2)	26(2)	33(2)	3.8(16)	3.2(18)	0.5(15)
C17	35(2)	37(2)	28(2)	6.8(17)	4.6(17)	12.6(18)
C18	18.7(15)	17.7(16)	29.9(19)	-1.9(15)	-1.5(13)	-0.9(13)

Table 4 Bond Lengths for 283.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C15	1.905(4)	C3	C4	1.384(5)
S2	O3	1.428(3)	C3	C6	1.387(6)
S2	C	1.782(4)	N13	C18	1.489(5)
S2	O9	1.432(3)	C4	C12	1.380(5)
S2	N13	1.611(3)	C5	C10	1.402(5)
O4	C2	1.216(5)	C5	C13	1.396(6)
O5	N6	1.228(5)	C7	C12	1.379(6)
N6	O10	1.220(5)	C8	C10	1.380(6)
N6	C4	1.473(5)	C8	C15	1.378(6)
C	C6	1.387(5)	C9	C18	1.537(5)
C	C7	1.390(5)	C11	C17	1.520(7)
C1	N13	1.478(5)	C13	C14	1.382(7)
C1	C11	1.514(6)	C14	C15	1.379(6)
C2	C5	1.492(6)	C16	C17	1.530(6)
C2	C9	1.515(5)	C16	C18	1.525(6)

Table 5 Bond Angles for 283.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
O3	S2	C	106.78(17)	C12	C4	C3	122.3(4)
O3	S2	O9	119.16(17)	C10	C5	C2	123.5(4)
O3	S2	N13	108.61(16)	C13	C5	C2	118.2(3)
O9	S2	C	106.19(18)	C13	C5	C10	118.2(4)
O9	S2	N13	107.93(17)	C	C6	C3	120.0(4)
N13	S2	C	107.64(17)	C12	C7	C	119.4(4)
O5	N6	C4	117.6(4)	C15	C8	C10	119.3(4)
O10	N6	O5	124.1(4)	C2	C9	C18	113.8(3)
O10	N6	C4	118.3(3)	C8	C10	C5	120.6(4)
C6	C	S2	119.4(3)	C1	C11	C17	110.8(4)
C6	C	C7	120.8(4)	C7	C12	C4	119.3(4)
C7	C	S2	119.8(3)	C14	C13	C5	121.5(4)
N13	C1	C11	109.6(3)	C15	C14	C13	118.5(4)
O4	C2	C5	120.2(4)	C8	C15	Br1	119.3(3)
O4	C2	C9	120.8(4)	C8	C15	C14	121.8(4)
C5	C2	C9	119.0(3)	C14	C15	Br1	118.9(3)
C4	C3	C6	118.2(4)	C18	C16	C17	112.2(3)
C1	N13	S2	120.1(3)	C11	C17	C16	109.4(3)
C1	N13	C18	116.2(3)	N13	C18	C9	112.3(3)
C18	N13	S2	120.4(2)	N13	C18	C16	107.6(3)

C3	C4	N6	119.1(3)	C16	C18	C9	112.9(3)
C12	C4	N6	118.7(3)				

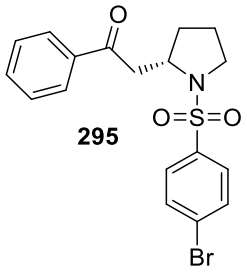
Table 6 Torsion Angles for 283.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
S2	C	C6	C3	-179.4(3)	C2	C9	C18	N13	95.7(4)
S2	C	C7	C12	-179.5(3)	C2	C9	C18	C16	-142.4(3)
S2	N13	C18	C9	-90.0(3)	C3	C4	C12	C7	1.1(6)
S2	N13	C18	C16	145.1(3)	N13	S2	C	C6	-129.7(3)
O3	S2	C	C6	-13.2(3)	N13	S2	C	C7	50.7(3)
O3	S2	C	C7	167.2(3)	N13	C1	C11	C17	-55.5(4)
O3	S2	N13	C1	164.1(3)	C4	C3	C6	C	-0.7(6)
O3	S2	N13	C18	-37.0(3)	C5	C2	C9	C18	-157.5(3)
O4	C2	C5	C10	177.4(4)	C5	C13	C14	C15	0.1(6)
O4	C2	C5	C13	-4.7(5)	C6	C	C7	C12	0.9(5)
O4	C2	C9	C18	23.5(5)	C6	C3	C4	N6	-179.7(3)
O5	N6	C4	C3	-5.0(5)	C6	C3	C4	C12	0.0(6)
O5	N6	C4	C12	175.3(4)	C7	C	C6	C3	0.2(6)
N6	C4	C12	C7	-179.2(3)	C9	C2	C5	C10	-1.6(5)
C	S2	N13	C1	-80.6(3)	C9	C2	C5	C13	176.3(3)
C	S2	N13	C18	78.2(3)	C10	C5	C13	C14	0.7(6)
C	C7	C12	C4	-1.6(6)	C10	C8	C15	Br1	-178.7(3)
C1	N13	C18	C9	69.6(4)	C10	C8	C15	C14	1.6(6)
C1	N13	C18	C16	-55.3(4)	C11	C1	N13	S2	-143.8(3)
C1	C11	C17	C16	57.1(5)	C11	C1	N13	C18	56.5(4)
O9	S2	C	C6	114.9(3)	C13	C5	C10	C8	-0.3(6)
O9	S2	C	C7	-64.7(3)	C13	C14	C15	Br1	179.1(3)
O9	S2	N13	C1	33.6(3)	C13	C14	C15	C8	-1.2(6)
O9	S2	N13	C18	-167.5(3)	C15	C8	C10	C5	-0.8(6)
O10	N6	C4	C3	175.4(4)	C17	C16	C18	N13	54.6(4)
O10	N6	C4	C12	-4.3(5)	C17	C16	C18	C9	-69.9(4)
C2	C5	C10	C8	177.6(4)	C18	C16	C17	C11	-57.6(5)
C2	C5	C13	C14	-177.3(4)					

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 283.

Atom	*x*	*y*	*z*	U(eq)
H1A	-1261	4925	2303	34
H1B	-1198	3911	1954	34
H3	3848	3153	4505	34
H6	4869	3061	3540	31
H7	-1403	3897	3080	30
H8	-1501	6875	4514	35
H9A	2720	6623	2672	31
H9B	968	5787	2849	31
H10	-55	6571	3609	33
H11A	-2054	5265	1344	41
H11B	195	4719	1146	41
H12	-2385	4039	4041	30
H13	5556	5431	4325	34
H14	4084	5720	5230	38
H16A	3972	5477	1395	36
H16B	4035	6492	1740	36
H17A	66	6511	1793	40
H17B	819	6432	1138	40
H18	4885	5084	2319	27

X-Ray data for (*S*)-2-(1-((4-bromophenyl)sulfonyl)pyrrolidin-2-yl)-1-phenylethan-1-one **295**



295

Table 1 Crystal data and structure refinement for 295.

Identification code	**295**
Empirical formula	$C_{18}H_{18}NO_3SBr$
Formula weight	408.30
Temperature/K	120(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	10.1884(6)
b/Å	12.2084(8)
c/Å	13.9081(8)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1729.94(18)
Z	4
ρ_{calc}g/cm^3	1.568
μ/mm^{-1}	4.505
F(000)	832.0
Crystal size/mm^3	0.4709 × 0.2804 × 0.2032
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.64 to 148.276
Index ranges	-12 ≤ h ≤ 8, -13 ≤ k ≤ 15, -17 ≤ l ≤ 11
Reflections collected	6359
Independent reflections	3391 [R_{int} = 0.0257, R_{sigma} = 0.0315]
Data/restraints/parameters	3391/0/217
Goodness-of-fit on F^2	1.058
Final R indexes [I>=2σ (I)]	R_1 = 0.0254, wR_2 = 0.0681
Final R indexes [all data]	R_1 = 0.0258, wR_2 = 0.0684
Largest diff. peak/hole / e Å^{-3}	0.30/-0.50
Flack parameter	0.000(12)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 295. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	7459.5(6)	4377.8(6)	2994.1(5)	17.89(15)
N2	7570(2)	4351(2)	4156.0(16)	19.2(5)
C3	6409(3)	3967(2)	4727(2)	20.8(6)
C4	7045(4)	3294(3)	5534(2)	29.9(7)

C5	8428(4)	3766(3)	5630(2)	33.7(8)
C6	8830(3)	3995(3)	4599(2)	24.6(7)
O7	8599(2)	4933.0(19)	2637.4(16)	24.7(5)
O8	6170(2)	4786.4(19)	2769.7(17)	24.9(5)
C9	7542(3)	3009(2)	2583.0(19)	18.0(5)
C10	8759(3)	2556(3)	2367(2)	21.3(6)
C11	8835(3)	1461(3)	2100(2)	24.3(6)
C12	7690(3)	851(2)	2057(2)	21.9(6)
C13	6469(3)	1295(3)	2258(2)	22.8(6)
C14	6397(3)	2398(3)	2524(2)	21.6(6)
Br15	7789.7(3)	-651.8(3)	1720.1(2)	28.01(11)
C16	5634(3)	4956(3)	5069(2)	23.2(6)
C17	4255(3)	4674(3)	5397(2)	22.0(6)
O18	3912(2)	3727(2)	5478.4(18)	27.4(5)
C19	3326(3)	5599(3)	5593.6(19)	20.7(6)
C20	3738(3)	6688(3)	5631(2)	25.9(7)
C21	2845(4)	7514(3)	5844(3)	30.2(7)
C22	1534(4)	7252(3)	6011(3)	31.7(8)
C23	1116(3)	6178(3)	5956(3)	29.3(7)
C24	1999(3)	5354(3)	5749(2)	24.6(7)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 295. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	18.3(3)	15.1(3)	20.3(3)	1.1(2)	0.6(2)	1.0(3)
N2	20.5(11)	17.8(11)	19.2(11)	-0.6(9)	-0.4(9)	1.6(10)
C3	23.1(13)	16.7(14)	22.5(14)	-1.0(11)	3.2(11)	0.3(11)
C4	37.0(19)	26.9(17)	25.8(14)	6.3(13)	4.9(14)	8.9(15)
C5	36.6(18)	41(2)	23.1(16)	-2.7(15)	-6.5(14)	10.7(16)
C6	22.4(14)	24.5(17)	26.9(15)	-2.3(12)	-4.3(12)	4.1(12)
O7	26.7(10)	20.2(11)	27.3(11)	0.5(9)	5.4(9)	-4.6(9)
O8	23.4(10)	24.0(11)	27.3(11)	3.4(9)	-1.9(9)	5.6(9)
C9	20.6(14)	17.1(13)	16.3(11)	0.8(10)	-0.1(10)	0.9(11)
C10	17.0(13)	22.3(15)	24.5(14)	-0.9(12)	-0.3(11)	-1.2(12)
C11	21.9(14)	24.0(16)	27.0(14)	-3.9(13)	0.5(12)	5.2(12)
C12	31.3(14)	17.2(14)	17.3(12)	-1.9(10)	-2.3(11)	0.2(12)
C13	22.5(14)	21.7(16)	24.3(15)	-1.1(13)	-2.3(11)	-3.5(12)
C14	17.9(13)	25.2(16)	21.8(14)	2.1(12)	-0.5(11)	-0.9(12)
Br15	39.99(19)	18.49(17)	25.55(16)	-4.29(12)	0.46(13)	-0.11(14)
C16	24.8(14)	17.9(16)	26.9(15)	0.0(12)	3.5(12)	1.3(12)
C17	25.8(14)	20.3(16)	19.8(13)	0.4(11)	0.1(11)	1.9(12)
O18	30.5(12)	18.7(12)	32.9(12)	0.4(9)	4.3(10)	-1.2(10)
C19	23.3(13)	21.8(14)	17.0(12)	-0.4(12)	-0.2(10)	0.0(13)
C20	24.9(15)	24.5(17)	28.3(15)	-0.3(14)	3.1(12)	-0.3(13)
C21	32.8(17)	19.6(15)	38.4(17)	2.7(13)	8.3(15)	1.0(15)
C22	29.9(16)	30.8(19)	34.2(17)	4.3(15)	4.3(14)	10.8(15)
C23	23.8(15)	36.2(19)	28.1(15)	4.9(14)	2.6(13)	1.8(15)
C24	25.2(14)	22.8(16)	25.7(14)	1.7(12)	2.2(12)	-4.0(12)

Table 4 Bond Lengths for 295.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	N2	1.620(2)	C11	C12	1.385(5)
S1	O7	1.433(2)	C12	C13	1.385(4)
S1	O8	1.440(2)	C12	Br15	1.897(3)
S1	C9	1.768(3)	C13	C14	1.399(5)
N2	C3	1.499(4)	C16	C17	1.517(4)
N2	C6	1.489(4)	C17	O18	1.213(4)
C3	C4	1.534(4)	C17	C19	1.499(4)
C3	C16	1.520(4)	C19	C20	1.395(5)
C4	C5	1.528(5)	C19	C24	1.402(4)
C5	C6	1.517(5)	C20	C21	1.391(5)
C9	C10	1.391(4)	C21	C22	1.393(5)
C9	C14	1.387(4)	C22	C23	1.380(5)
C10	C11	1.389(5)	C23	C24	1.380(5)

Table 5 Bond Angles for 295.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
N2	S1	C9	107.48(13)	C12	C11	C10	118.8(3)
O7	S1	N2	107.38(13)	C11	C12	C13	122.5(3)
O7	S1	O8	120.04(14)	C11	C12	Br15	119.1(2)
O7	S1	C9	107.25(14)	C13	C12	Br15	118.4(2)
O8	S1	N2	106.64(13)	C12	C13	C14	118.5(3)
O8	S1	C9	107.49(13)	C9	C14	C13	119.3(3)
C3	N2	S1	118.67(19)	C17	C16	C3	113.3(3)
C6	N2	S1	118.6(2)	O18	C17	C16	120.8(3)
C6	N2	C3	111.7(2)	O18	C17	C19	121.3(3)
N2	C3	C4	102.8(2)	C19	C17	C16	117.9(3)
N2	C3	C16	109.1(2)	C20	C19	C17	122.4(3)
C16	C3	C4	114.6(3)	C20	C19	C24	119.2(3)
C5	C4	C3	104.6(3)	C24	C19	C17	118.4(3)
C6	C5	C4	103.7(3)	C21	C20	C19	120.2(3)
N2	C6	C5	102.2(3)	C20	C21	C22	119.7(3)
C10	C9	S1	119.2(2)	C23	C22	C21	120.3(3)
C14	C9	S1	119.2(2)	C24	C23	C22	120.2(3)
C14	C9	C10	121.5(3)	C23	C24	C19	120.4(3)
C11	C10	C9	119.3(3)				

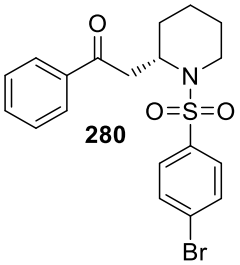
Table 6 Torsion Angles for 295.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
S1	N2	C3	C4	140.8(2)	O8	S1	C9	C14	-27.2(3)
S1	N2	C3	C16	-97.2(3)	C9	S1	N2	C3	-73.9(2)
S1	N2	C6	C5	-164.3(2)	C9	S1	N2	C6	67.0(2)
S1	C9	C10	C11	176.1(2)	C9	C10	C11	C12	0.0(5)
S1	C9	C14	C13	-176.0(2)	C10	C9	C14	C13	1.1(4)
N2	S1	C9	C10	-89.9(2)	C10	C11	C12	C13	0.9(5)
N2	S1	C9	C14	87.2(2)	C10	C11	C12	Br15	-178.6(2)
N2	C3	C4	C5	25.1(3)	C11	C12	C13	C14	-0.7(5)
N2	C3	C16	C17	163.3(2)	C12	C13	C14	C9	-0.3(5)
C3	N2	C6	C5	-20.7(3)	C14	C9	C10	C11	-1.0(4)
C3	C4	C5	C6	-38.5(3)	Br15	C12	C13	C14	178.8(2)
C3	C16	C17	O18	8.6(4)	C16	C3	C4	C5	-93.1(3)
C3	C16	C17	C19	-170.0(3)	C16	C17	C19	C20	-10.1(4)
C4	C3	C16	C17	-82.1(3)	C16	C17	C19	C24	170.1(3)
C4	C5	C6	N2	35.7(3)	C17	C19	C20	C21	-178.1(3)
C6	N2	C3	C4	-2.7(3)	C17	C19	C24	C23	178.4(3)
C6	N2	C3	C16	119.3(3)	O18	C17	C19	C20	171.2(3)
O7	S1	N2	C3	171.0(2)	O18	C17	C19	C24	-8.5(4)
O7	S1	N2	C6	-48.1(3)	C19	C20	C21	C22	-0.6(5)
O7	S1	C9	C10	25.3(3)	C20	C19	C24	C23	-1.3(5)
O7	S1	C9	C14	-157.6(2)	C20	C21	C22	C23	-0.7(6)
O8	S1	N2	C3	41.1(2)	C21	C22	C23	C24	1.1(6)
O8	S1	N2	C6	-177.9(2)	C22	C23	C24	C19	0.0(5)
O8	S1	C9	C10	155.6(2)	C24	C19	C20	C21	1.6(5)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 295.

Atom	*x*	*y*	*z*	U(eq)
H3	5840	3485	4321	25
H4A	6550	3378	6142	36
H4B	7077	2508	5361	36
H5A	8422	4447	6016	40
H5B	9028	3231	5934	40
H6A	9499	4582	4566	30
H6B	9176	3328	4283	30
H10	9531	2990	2402	26
H11	9657	1136	1949	29
H13	5698	860	2216	27
H14	5573	2725	2664	26
H16A	6111	5305	5608	28
H16B	5581	5496	4539	28
H20	4631	6866	5510	31
H21	3127	8255	5875	36
H22	925	7814	6164	38
H23	218	6006	6060	35
H24	1707	4616	5712	29

X-Ray data for (S)-2-(1-((4-bromophenyl)sulfonyl)piperidin-2-yl)-1-phenylethan-1-one **280**



280

Table 1 Crystal data and structure refinement for 280.

Identification code	**280**
Empirical formula	$C_{19}H_{20}NO_3SBr$
Formula weight	422.33
Temperature/K	120(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	10.2690(2)
b/Å	12.6538(3)
c/Å	14.0743(3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1828.85(7)
Z	4
ρ_{calc}g/cm^3	1.534
μ/mm^{-1}	4.282
F(000)	864.0
Crystal size/mm^3	0.214 × 0.156 × 0.129
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.398 to 154.434
Index ranges	-11 ≤ h ≤ 12, -15 ≤ k ≤ 15, -17 ≤ l ≤ 17
Reflections collected	9954
Independent reflections	3728 [R_{int} = 0.0280, R_{sigma} = 0.0290]
Data/restraints/parameters	3728/0/226
Goodness-of-fit on F^2	1.114
Final R indexes [I>=2σ (I)]	R_1 = 0.0331, wR_2 = 0.0849
Final R indexes [all data]	R_1 = 0.0359, wR_2 = 0.0890
Largest diff. peak/hole / e Å^{-3}	0.42/-0.51
Flack parameter	-0.032(10)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 280. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br16	7052.0(6)	5468.0(4)	6741.2(4)	59.66(18)
S7	7644.1(9)	677.4(7)	8144.0(7)	37.8(2)
O19	11398(3)	1082(2)	10369(3)	52.5(8)
O9	6510(3)	125(2)	7833(2)	48.5(8)
O8	8901(3)	332(3)	7838(2)	51.9(8)

N1	7629(3)	680(3)	9285(2)	43.0(8)
C21	13048(4)	-606(4)	10742(3)	49.8(10)
C12	8461(4)	3652(4)	7323(4)	47.2(11)
C6	6461(4)	459(4)	9847(3)	49.5(10)
C17	9455(4)	68(3)	10278(4)	43.1(10)
C10	7491(4)	2001(3)	7754(3)	35.7(8)
C18	10902(4)	209(3)	10424(3)	39.2(9)
C4	7279(5)	1662(4)	11111(4)	54.1(11)
C20	11720(4)	-738(3)	10636(3)	39.9(9)
C11	8586(4)	2615(4)	7618(3)	44.8(10)
C13	7226(5)	4048(3)	7163(3)	43.8(9)
C2	8808(4)	1029(3)	9808(3)	39.7(9)
C24	11994(6)	-2581(4)	11001(4)	61.3(13)
C15	6262(4)	2406(4)	7587(3)	43.1(10)
C14	6126(5)	3448(4)	7289(4)	48.6(11)
C23	13319(5)	-2433(4)	11111(4)	61.3(14)
C25	11194(5)	-1740(4)	10768(4)	49.9(11)
C22	13845(5)	-1444(5)	10975(4)	60.9(14)
C5	6116(5)	1368(4)	10494(4)	55.4(13)
C3	8451(5)	1915(3)	10492(3)	46.1(10)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 280. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br16	79.4(4)	38.5(3)	61.1(3)	10.6(2)	0.4(3)	0.4(2)
S7	35.7(4)	30.2(4)	47.5(5)	-4.1(4)	-1.6(4)	1.9(3)
O19	44.3(17)	32.1(16)	81(2)	9.9(16)	-3.1(17)	-3.9(14)
O9	49.6(17)	35.8(16)	60.0(18)	-7.0(14)	-8.9(14)	-7.3(13)
O8	45.3(17)	45.4(18)	64.9(19)	-6.4(16)	8.5(14)	13.4(15)
N1	34.7(17)	45(2)	48.9(19)	6.9(16)	-3.3(14)	-5.9(15)
C21	41(2)	46(3)	62(3)	5(2)	-0.5(19)	-2(2)
C12	38(2)	45(3)	58(3)	5(2)	3(2)	-6.4(19)
C6	43(2)	43(2)	62(3)	3(2)	4.7(19)	-4(2)
C17	41(2)	30(2)	59(3)	2.2(19)	-3.8(19)	0.5(17)
C10	34(2)	33(2)	39.8(19)	-2.7(16)	-0.4(15)	-0.3(15)
C18	41(2)	32(2)	44(2)	1.2(17)	-0.8(17)	-0.5(17)
C4	63(3)	40(2)	60(3)	-7(2)	5(2)	2(2)
C20	43(2)	35(2)	42(2)	1.8(17)	-0.5(17)	2.6(17)
C11	36(2)	45(3)	53(3)	0(2)	3.6(18)	-2.0(19)
C13	53(2)	34(2)	44(2)	0.2(17)	0(2)	0(2)
C2	35.0(19)	34(2)	50(2)	3.8(18)	-6.6(17)	-2.5(16)
C24	67(3)	34(2)	84(4)	4(2)	-5(3)	2(2)
C15	33(2)	42(2)	55(3)	4.9(19)	-1.1(18)	-0.3(18)
C14	42(2)	44(3)	60(3)	7(2)	-2(2)	5(2)
C23	56(3)	50(3)	78(4)	2(3)	-3(3)	19(2)
C25	43(2)	36(2)	71(3)	1(2)	-5(2)	0.1(19)
C22	44(3)	63(3)	76(4)	10(3)	-3(2)	10(2)
C5	44(3)	46(3)	77(4)	-2(2)	12(2)	1(2)
C3	50(2)	30(2)	59(3)	1.0(19)	-8(2)	-0.1(18)

Table 4 Bond Lengths for 280.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br16	C13	1.900(4)	C17	C2	1.536(6)
S7	O9	1.427(3)	C10	C11	1.380(6)
S7	O8	1.429(3)	C10	C15	1.381(5)
S7	N1	1.606(4)	C18	C20	1.494(6)
S7	C10	1.770(4)	C4	C5	1.522(7)
O19	C18	1.219(5)	C4	C3	1.520(7)
N1	C6	1.463(5)	C20	C25	1.391(6)
N1	C2	1.485(5)	C13	C14	1.373(6)
C21	C20	1.382(6)	C2	C3	1.521(6)
C21	C22	1.379(7)	C24	C23	1.382(8)
C12	C11	1.383(7)	C24	C25	1.383(7)
C12	C13	1.382(6)	C15	C14	1.391(6)
C6	C5	1.509(7)	C23	C22	1.377(8)
C17	C18	1.510(6)			

Table 5 Bond Angles for 280.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
O9	S7	O8	119.66(19)	C3	C4	C5	110.2(4)
O9	S7	N1	107.47(19)	C21	C20	C18	118.7(4)
O9	S7	C10	107.21(19)	C21	C20	C25	118.6(4)
O8	S7	N1	108.1(2)	C25	C20	C18	122.7(4)
O8	S7	C10	106.0(2)	C10	C11	C12	120.0(4)
N1	S7	C10	107.90(19)	C12	C13	Br16	118.7(3)
C6	N1	S7	123.2(3)	C14	C13	Br16	119.1(3)
C6	N1	C2	117.2(3)	C14	C13	C12	122.2(4)
C2	N1	S7	119.3(3)	N1	C2	C17	109.3(3)
C22	C21	C20	121.2(5)	N1	C2	C3	109.7(3)
C13	C12	C11	118.6(4)	C3	C2	C17	114.5(4)
N1	C6	C5	111.9(4)	C23	C24	C25	120.5(5)
C18	C17	C2	112.9(3)	C10	C15	C14	119.7(4)
C11	C10	S7	120.2(3)	C13	C14	C15	118.7(4)
C11	C10	C15	120.8(4)	C22	C23	C24	119.6(5)
C15	C10	S7	119.0(3)	C24	C25	C20	120.1(4)
O19	C18	C17	120.7(4)	C23	C22	C21	119.9(5)
O19	C18	C20	120.3(4)	C6	C5	C4	110.2(4)
C20	C18	C17	119.0(4)	C4	C3	C2	113.5(4)

Table 6 Torsion Angles for 280.

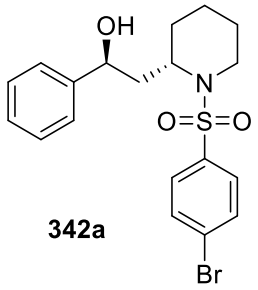
A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
Br16	C13	C14	C15	179.2(4)	C17	C18	C20	C25	-4.4(6)
S7	N1	C6	C5	-121.3(4)	C17	C2	C3	C4	-73.5(5)
S7	N1	C2	C17	-108.6(4)	C10	S7	N1	C6	97.8(4)
S7	N1	C2	C3	125.1(3)	C10	S7	N1	C2	-75.7(3)
S7	C10	C11	C12	-179.3(4)	C10	C15	C14	C13	0.4(7)
S7	C10	C15	C14	179.3(4)	C18	C17	C2	N1	153.2(4)
O19	C18	C20	C21	-1.7(6)	C18	C17	C2	C3	-83.3(5)
O19	C18	C20	C25	175.6(5)	C18	C20	C25	C24	-177.4(5)
O9	S7	N1	C6	-17.5(4)	C20	C21	C22	C23	-0.4(9)
O9	S7	N1	C2	169.0(3)	C11	C12	C13	Br16	-179.1(4)
O9	S7	C10	C11	-155.2(4)	C11	C12	C13	C14	0.2(7)
O9	S7	C10	C15	24.7(4)	C11	C10	C15	C14	-0.8(7)
O8	S7	N1	C6	-148.0(4)	C13	C12	C11	C10	-0.5(7)
O8	S7	N1	C2	38.6(4)	C2	N1	C6	C5	52.3(5)
O8	S7	C10	C11	-26.3(4)	C2	C17	C18	O19	15.6(7)
O8	S7	C10	C15	153.6(4)	C2	C17	C18	C20	-164.4(4)
N1	S7	C10	C11	89.3(4)	C24	C23	C22	C21	0.9(9)
N1	S7	C10	C15	-90.8(4)	C15	C10	C11	C12	0.9(7)
N1	C6	C5	C4	-54.2(6)	C23	C24	C25	C20	0.6(9)
N1	C2	C3	C4	49.8(5)	C25	C24	C23	C22	-1.0(9)
C21	C20	C25	C24	-0.1(7)	C22	C21	C20	C18	177.4(5)
C12	C13	C14	C15	-0.1(7)	C22	C21	C20	C25	0.0(7)
C6	N1	C2	C17	77.5(5)	C5	C4	C3	C2	-55.4(5)
C6	N1	C2	C3	-48.8(5)	C3	C4	C5	C6	56.1(5)
C17	C18	C20	C21	178.3(4)					

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 280.

Atom	*x*	*y*	*z*	U(eq)
H21	13419	75	10653	60
H12	9207	4084	7233	57
H6A	6609	-183	10234	59
H6B	5722	318	9414	59
H17A	9038	-62	10902	52
H17B	9305	-563	9877	52
H4A	7059	2283	11507	65
H4B	7492	1067	11541	65
H11	9426	2324	7726	54
H2	9435	1323	9333	48
H24	11630	-3265	11085	74
H15	5515	1975	7675	52
H14	5287	3739	7175	58
H23	13863	-3011	11281	74
H25	10283	-1848	10699	60
H22	14757	-1339	11041	73
H5A	5373	1166	10904	66
H5B	5851	1986	10108	66

H3A	9208	2063	10907	55
H3B	8265	2563	10122	55

X-Ray data for (*S*)-2-((*S*)-1-((4-bromophenyl)sulfonyl)piperidin-2-yl)-1-phenylethan-1-ol **342a**



342a

Table 1 Crystal data and structure refinement for 342a.

Identification code	342a
Empirical formula	$C_{19}H_{22}NO_3SBr$
Formula weight	424.34
Temperature/K	120(2)
Crystal system	monoclinic
Space group	Cc
a/Å	8.7445(5)
b/Å	19.3497(11)
c/Å	11.3150(7)
α/°	90
β/°	95.088(5)
γ/°	90
Volume/Å^3	1907.0(2)
Z	4
ρ_{calc}g/cm^3	1.478
μ/mm^{-1}	4.106
F(000)	872.0
Crystal size/mm^3	0.3896 × 0.1809 × 0.1639
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	11.14 to 133.006
Index ranges	-7 ≤ h ≤ 10, -22 ≤ k ≤ 22, -13 ≤ l ≤ 13
Reflections collected	8488
Independent reflections	2499 [R_{int} = 0.0280, R_{sigma} = 0.0231]
Data/restraints/parameters	2499/2/229
Goodness-of-fit on F^2	1.087
Final R indexes [I>=2σ (I)]	R_1 = 0.0243, wR_2 = 0.0636
Final R indexes [all data]	R_1 = 0.0245, wR_2 = 0.0638
Largest diff. peak/hole / e Å^{-3}	0.21/-0.46
Flack parameter	-0.024(12)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 342a. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
N1	5698(3)	2962.2(14)	1768(2)	21.6(5)
C2	7364(3)	2833.5(15)	1708(3)	19.7(6)
C3	7519(4)	2112.2(17)	1165(3)	29.1(7)
C4	6579(5)	2052.8(19)	-32(3)	34.5(8)
C5	4909(5)	2231(2)	76(3)	42.5(10)
C6	4747(4)	2936(2)	630(3)	32.5(8)
S7	5142.3(8)	3371.3(4)	2895.5(6)	24.72(18)
O8	5960(3)	3103.9(14)	3944(2)	32.6(6)
O9	3494(3)	3348.5(16)	2776(3)	40.0(7)
C10	5663(4)	4250.1(18)	2780(3)	25.4(7)
C11	6913(4)	4510.5(19)	3482(3)	30.3(7)
C12	7359(5)	5190.7(19)	3359(3)	32.8(8)
C13	6529(4)	5598.8(19)	2518(3)	31.2(8)
C14	5294(4)	5343(2)	1807(3)	33.6(8)
C15	4851(4)	4667.1(19)	1939(3)	31.1(8)
Br16	7116.6(4)	6540.3(2)	2357.2(4)	44.36(14)
C17	8117(4)	3402.7(15)	1002(3)	18.2(6)
C18	9855(4)	3357.7(16)	1141(3)	20.7(6)
O19	10286(3)	3392.2(15)	2388(2)	33.7(6)
C20	10562(3)	3946.9(16)	487(3)	19.4(6)
C21	10506(4)	4617.8(17)	927(3)	25.9(7)
C22	11116(4)	5164.1(17)	323(3)	29.2(7)
C23	11778(4)	5044.1(19)	-729(3)	31.7(8)
C24	11835(4)	4378.4(18)	-1172(3)	29.6(7)
C25	11224(4)	3833.1(18)	-568(3)	24.7(6)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 342a. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	15.0(12)	29.9(14)	19.9(12)	-2.6(10)	1.1(10)	-3.3(10)
C2	14.1(14)	24.4(16)	21.1(14)	4.0(12)	3.6(11)	-1.5(12)
C3	31.6(18)	19.7(16)	38.1(19)	7.0(13)	15.5(15)	-2.1(13)
C4	50(2)	26.1(17)	30.8(18)	-7.8(13)	19.4(16)	-11.8(15)
C5	36(2)	64(3)	27.8(19)	-15.4(17)	2.6(15)	-22.9(19)
C6	16.9(16)	57(2)	22.6(16)	0.2(15)	-3.5(13)	-4.8(15)
S7	19.6(4)	34.3(4)	21.3(4)	-1.4(3)	8.0(3)	-2.2(3)
O8	40.1(14)	36.7(15)	21.5(11)	0.8(10)	6.2(10)	-4.6(11)
O9	22.9(13)	53.7(18)	45.8(16)	-2.9(12)	16.9(12)	-4.6(11)
C10	22.8(15)	31.3(18)	23.3(15)	-5.1(12)	7.3(12)	4.1(14)
C11	33.7(19)	32.1(18)	24.3(16)	-2.8(13)	-2.5(14)	5.2(15)
C12	37(2)	33.9(19)	26.2(18)	-6.2(14)	-3.2(14)	0.3(16)
C13	40(2)	30.4(17)	23.8(16)	-4.1(13)	5.0(14)	3.8(15)
C14	32(2)	37(2)	30.9(19)	1.6(14)	-0.8(15)	10.4(15)
C15	23.7(17)	39(2)	29.5(18)	-3.6(14)	-1.2(14)	5.7(15)
Br16	65.0(3)	31.40(19)	36.2(2)	1.60(16)	1.68(17)	-1.7(2)

C17	16.4(14)	18.4(15)	19.5(15)	3(1)	0.7(11)	-0.2(11)
C18	18.8(16)	23.2(16)	20.5(15)	5.7(11)	3.6(12)	0.4(12)
O19	17.6(12)	57.2(18)	25.2(13)	16.3(11)	-3.8(10)	-10.0(11)
C20	13.7(12)	22.7(15)	21.5(14)	4.0(11)	0.5(11)	-3.5(11)
C21	28.0(17)	29.2(17)	20.6(15)	-0.6(12)	3.9(13)	-2.5(14)
C22	35.2(17)	22.7(16)	29.6(18)	-1.9(13)	2.9(15)	-9.6(14)
C23	36(2)	28.6(18)	31.1(18)	7.4(13)	4.9(15)	-7.8(15)
C24	32.8(17)	34.4(19)	22.5(16)	1.8(13)	7.0(13)	-4.2(15)
C25	25.6(16)	25.3(16)	24.1(15)	-0.5(12)	7.5(12)	-1.1(12)

Table 4 Bond Lengths for 342a.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C2	1.485(4)	C12	C13	1.390(5)
N1	C6	1.470(4)	C13	C14	1.380(5)
N1	S7	1.613(3)	C13	Br16	1.906(4)
C2	C3	1.536(4)	C14	C15	1.376(6)
C2	C17	1.542(4)	C17	C18	1.517(4)
C3	C4	1.524(5)	C18	O19	1.430(4)
C4	C5	1.516(6)	C18	C20	1.520(4)
C5	C6	1.514(6)	C20	C21	1.393(5)
S7	O8	1.427(3)	C20	C25	1.390(4)
S7	O9	1.437(3)	C21	C22	1.390(5)
S7	C10	1.768(4)	C22	C23	1.388(5)
C10	C11	1.388(5)	C23	C24	1.385(5)
C10	C15	1.393(5)	C24	C25	1.389(5)
C11	C12	1.383(5)			

Table 5 Bond Angles for 342a.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C2	N1	S7	119.0(2)	C12	C11	C10	120.1(3)
C6	N1	C2	115.8(2)	C11	C12	C13	118.3(3)
C6	N1	S7	121.5(2)	C12	C13	Br16	118.6(3)
N1	C2	C3	107.0(2)	C14	C13	C12	122.0(3)
N1	C2	C17	111.7(2)	C14	C13	Br16	119.4(3)
C3	C2	C17	112.6(2)	C15	C14	C13	119.4(3)
C4	C3	C2	111.4(3)	C14	C15	C10	119.6(3)
C5	C4	C3	111.0(3)	C18	C17	C2	112.1(2)
C6	C5	C4	111.4(3)	C17	C18	C20	110.7(3)
N1	C6	C5	109.1(3)	O19	C18	C17	105.9(3)
N1	S7	C10	108.41(15)	O19	C18	C20	111.3(3)
O8	S7	N1	108.40(15)	C21	C20	C18	119.8(3)
O8	S7	O9	119.47(16)	C25	C20	C18	121.2(3)
O8	S7	C10	107.26(16)	C25	C20	C21	119.0(3)
O9	S7	N1	106.32(15)	C22	C21	C20	120.4(3)
O9	S7	C10	106.58(17)	C23	C22	C21	120.1(3)
C11	C10	S7	120.1(3)	C24	C23	C22	119.8(3)
C11	C10	C15	120.6(3)	C23	C24	C25	120.0(3)

C15	C10	S7	119.2(3)	C24	C25	C20	120.6(3)

Table 6 Hydrogen Bonds for 342a.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O19	H19	O9[1]	0.78(6)	2.05(6)	2.801(4)	161(6)

[1]1+X,+Y,+Z

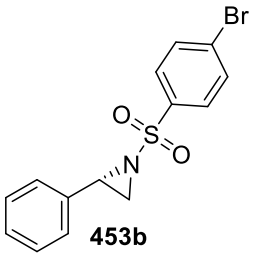
Table 7 Torsion Angles for 342a.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
N1	C2	C3	C4	55.3(3)	O8	S7	C10	C15	-169.9(3)
N1	C2	C17	C18	168.6(2)	O9	S7	C10	C11	142.1(3)
N1	S7	C10	C11	-103.8(3)	O9	S7	C10	C15	-40.9(3)
N1	S7	C10	C15	73.2(3)	C10	C11	C12	C13	-0.2(5)
C2	N1	C6	C5	58.8(4)	C11	C10	C15	C14	0.0(5)
C2	N1	S7	O8	-44.0(3)	C11	C12	C13	C14	-0.4(6)
C2	N1	S7	O9	-173.6(2)	C11	C12	C13	Br16	179.1(3)
C2	N1	S7	C10	72.1(3)	C12	C13	C14	C15	0.9(5)
C2	C3	C4	C5	-55.7(4)	C13	C14	C15	C10	-0.6(5)
C2	C17	C18	O19	-56.4(3)	C15	C10	C11	C12	0.5(5)
C2	C17	C18	C20	-177.1(3)	Br16	C13	C14	C15	-178.7(3)
C3	C2	C17	C18	-71.0(3)	C17	C2	C3	C4	-67.8(3)
C3	C4	C5	C6	54.6(4)	C17	C18	C20	C21	71.4(4)
C4	C5	C6	N1	-54.2(4)	C17	C18	C20	C25	-106.5(4)
C6	N1	C2	C3	-58.8(4)	C18	C20	C21	C22	-178.5(3)
C6	N1	C2	C17	64.8(4)	C18	C20	C25	C24	178.5(3)
C6	N1	S7	O8	158.7(3)	O19	C18	C20	C21	-46.1(4)
C6	N1	S7	O9	29.1(3)	O19	C18	C20	C25	136.1(3)
C6	N1	S7	C10	-85.2(3)	C20	C21	C22	C23	0.4(5)
S7	N1	C2	C3	142.6(2)	C21	C20	C25	C24	0.7(5)
S7	N1	C2	C17	-93.7(3)	C21	C22	C23	C24	-0.3(6)
S7	N1	C6	C5	-143.2(3)	C22	C23	C24	C25	0.3(5)
S7	C10	C11	C12	177.4(3)	C23	C24	C25	C20	-0.5(5)
S7	C10	C15	C14	-177.0(3)	C25	C20	C21	C22	-0.6(5)
O8	S7	C10	C11	13.1(3)					

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 342a.

Atom	*x*	*y*	*z*	U(eq)
H2	7874	2828	2535	24
H3A	8613	2020	1061	35
H3B	7166	1760	1713	35
H4A	7004	2370	-607	41
H4B	6652	1575	-336	41
H5A	4342	2224	-721	51
H5B	4448	1878	569	51
H6A	5081	3299	92	39
H6B	3658	3022	762	39
H11	7464	4221	4049	36
H12	8210	5375	3838	39
H14	4755	5631	1231	40
H15	3996	4486	1461	37
H17A	7752	3360	152	22
H17B	7795	3861	1280	22
H18	10191	2906	823	25
H19	11140(70)	3280(30)	2530(50)	51
H21	10047	4703	1643	31
H22	11080	5620	631	35
H23	12191	5418	-1143	38
H24	12293	4294	-1889	36
H25	11258	3378	-880	30

X-Ray data for (*S*)-1-((4-bromophenyl)sulfonyl)-2-phenylaziridine **453b**



453b

Table 1 Crystal data and structure refinement for 453b.

Identification code	**453b**
Empirical formula	$C_{14}H_{12}NO_2SBr$
Formula weight	338.22
Temperature/K	120(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	5.4440(9)
b/Å	8.7184(17)
c/Å	28.533(6)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1354.3(4)
Z	4
ρ_{calc}g/cm^3	1.659
μ/mm^{-1}	5.558
F(000)	680.0
Crystal size/mm^3	0.5379 × 0.246 × 0.1738
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.196 to 149.11
Index ranges	-5 ≤ h ≤ 6, -7 ≤ k ≤ 10, -35 ≤ l ≤ 33
Reflections collected	4842
Independent reflections	2686 [R_{int} = 0.0440, R_{sigma} = 0.0425]
Data/restraints/parameters	2686/0/172
Goodness-of-fit on F^2	1.070
Final R indexes [I>=2σ (I)]	R_1 = 0.0371, wR_2 = 0.0966
Final R indexes [all data]	R_1 = 0.0401, wR_2 = 0.1035
Largest diff. peak/hole / e Å^{-3}	0.46/-0.60
Flack parameter	0.01(2)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 453b. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	13015(2)	2011.3(14)	3944.9(4)	15.4(3)
N2	11733(9)	309(5)	3872.3(15)	16.0(9)
C3	9284(10)	148(6)	3661.6(17)	16.5(11)
C4	9698(11)	-245(6)	4166.8(17)	20.3(11)
O5	14636(7)	1851(4)	4336.9(12)	21.0(7)
O6	13977(7)	2410(4)	3495.8(12)	19.2(8)
C7	10747(10)	3364(5)	4093.8(17)	16.1(10)
C8	9476(11)	4122(6)	3738.1(18)	20.0(11)
C9	7710(11)	5192(6)	3855(2)	21.4(11)
C10	7215(12)	5473(6)	4324(2)	23.2(12)
C11	8477(11)	4721(6)	4680.0(19)	20.2(11)
C12	10210(11)	3651(5)	4566.0(17)	18.4(10)
Br13	4780.4(12)	6936.5(7)	4488.0(2)	30.7(2)
C14	9059(11)	-1031(6)	3283.8(17)	18.0(11)
C15	10799(11)	-2148(6)	3212.0(19)	21.7(11)
C16	10573(12)	-3223(7)	2856(2)	27.7(12)
C17	8518(13)	-3177(7)	2567(2)	29.8(13)
C18	6755(11)	-2061(7)	2631.7(19)	26.0(12)
C19	6988(12)	-993(7)	2993.4(19)	24.4(12)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 453b. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	16.4(6)	16.5(5)	13.4(5)	-1.8(4)	0.5(4)	-0.7(5)
N2	14(2)	16.4(19)	17(2)	-1.5(16)	1.0(17)	-0.4(18)
C3	18(3)	16(2)	15(2)	1.3(18)	1(2)	2(2)
C4	24(3)	20(2)	16(2)	-1.5(18)	2(2)	-2(2)
O5	19.4(19)	25.6(17)	18.1(17)	-2.8(14)	-3.1(14)	2.5(18)
O6	19.7(19)	25.0(18)	12.9(17)	-0.9(14)	3.7(14)	-1.0(15)
C7	20(3)	13(2)	15(2)	-1.7(16)	0.8(19)	-1.2(19)
C8	25(3)	18(2)	16(2)	1.4(19)	-1(2)	-4(2)
C9	22(3)	18(2)	24(3)	4(2)	-4(2)	2(2)
C10	31(3)	13(2)	26(3)	-3.7(19)	5(2)	-6(2)
C11	24(3)	18(2)	19(3)	-3.4(19)	2(2)	-5(2)
C12	23(3)	18(2)	13(2)	0.0(17)	0(2)	-1(2)
Br13	25.4(3)	22.0(3)	44.8(4)	-5.8(2)	3.0(3)	6.4(3)
C14	25(3)	15(2)	14(2)	0.8(18)	4(2)	-3(2)
C15	21(3)	22(3)	22(3)	-2(2)	0(2)	0(2)
C16	31(3)	22(3)	30(3)	-5(2)	12(2)	0(3)
C17	44(4)	27(3)	18(3)	-9(2)	9(2)	-10(3)
C18	24(3)	35(3)	19(3)	-6(2)	0(2)	-7(3)
C19	29(3)	26(3)	18(3)	-4(2)	4(2)	-1(3)

Table 4 Bond Lengths for 453b.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	N2	1.653(4)	C9	C10	1.385(8)
S1	O5	1.431(4)	C10	C11	1.391(8)
S1	O6	1.427(4)	C10	Br13	1.898(6)
S1	C7	1.760(5)	C11	C12	1.366(8)
N2	C3	1.469(7)	C14	C15	1.374(8)
N2	C4	1.472(7)	C14	C19	1.399(8)
C3	C4	1.499(7)	C15	C16	1.388(8)
C3	C14	1.495(7)	C16	C17	1.390(9)
C7	C8	1.395(7)	C17	C18	1.379(9)
C7	C12	1.401(7)	C18	C19	1.396(8)
C8	C9	1.381(8)			

Table 5 Bond Angles for 453b.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
N2	S1	C7	109.6(2)	C9	C8	C7	119.3(5)
O5	S1	N2	105.7(2)	C8	C9	C10	119.3(5)
O5	S1	C7	108.0(2)	C9	C10	C11	121.7(5)
O6	S1	N2	105.1(2)	C9	C10	Br13	119.5(4)
O6	S1	O5	120.0(2)	C11	C10	Br13	118.8(4)
O6	S1	C7	108.1(2)	C12	C11	C10	119.3(5)
C3	N2	S1	121.3(4)	C11	C12	C7	119.7(5)
C3	N2	C4	61.3(3)	C15	C14	C3	122.6(5)
C4	N2	S1	122.7(3)	C15	C14	C19	119.0(5)
N2	C3	C4	59.5(3)	C19	C14	C3	118.5(5)
N2	C3	C14	115.8(4)	C14	C15	C16	121.8(5)
C14	C3	C4	123.3(4)	C15	C16	C17	119.1(5)
N2	C4	C3	59.3(3)	C18	C17	C16	120.1(5)
C8	C7	S1	119.3(4)	C17	C18	C19	120.4(6)
C8	C7	C12	120.8(5)	C18	C19	C14	119.7(6)
C12	C7	S1	119.9(4)				

Table 6 Torsion Angles for 453b.

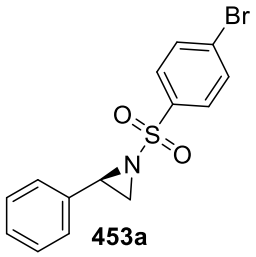
A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
S1	N2	C3	C4	-112.8(4)	O6	S1	C7	C8	26.9(5)
S1	N2	C3	C14	132.2(4)	O6	S1	C7	C12	-154.2(4)
S1	N2	C4	C3	110.7(5)	C7	S1	N2	C3	36.3(4)
S1	C7	C8	C9	-179.5(4)	C7	S1	N2	C4	-37.6(5)
S1	C7	C12	C11	178.9(4)	C7	C8	C9	C10	-0.9(8)
N2	S1	C7	C8	-87.2(5)	C8	C7	C12	C11	-2.2(8)
N2	S1	C7	C12	91.8(5)	C8	C9	C10	C11	0.9(8)
N2	C3	C14	C15	15.4(7)	C8	C9	C10	Br13	-179.8(4)
N2	C3	C14	C19	-164.7(5)	C9	C10	C11	C12	-1.5(8)
C3	C14	C15	C16	-179.3(5)	C10	C11	C12	C7	2.1(8)
C3	C14	C19	C18	178.7(5)	C12	C7	C8	C9	1.5(8)

C4	N2	C3	C14	-115.0(5)	Br13	C10	C11	C12	179.1(4)
C4	C3	C14	C15	-53.6(8)	C14	C3	C4	N2	102.6(6)
C4	C3	C14	C19	126.2(6)	C14	C15	C16	C17	-0.6(8)
O5	S1	N2	C3	152.5(4)	C15	C14	C19	C18	-1.4(8)
O5	S1	N2	C4	78.6(4)	C15	C16	C17	C18	0.9(8)
O5	S1	C7	C8	158.1(4)	C16	C17	C18	C19	-1.5(9)
O5	S1	C7	C12	-22.9(5)	C17	C18	C19	C14	1.8(9)
O6	S1	N2	C3	-79.7(4)	C19	C14	C15	C16	0.9(8)
O6	S1	N2	C4	-153.6(4)					

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 453b.

Atom	*x*	*y*	*z*	U(eq)
H3	8388	1130	3603	20
H4A	9029	462	4406	24
H4B	9689	-1342	4256	24
H8	9822	3904	3419	24
H9	6844	5729	3618	26
H11	8137	4949	4999	24
H12	11046	3103	4806	22
H15	12195	-2185	3411	26
H16	11805	-3980	2810	33
H17	8328	-3915	2325	36
H18	5373	-2019	2429	31
H19	5747	-244	3042	29

X-Ray data for (*R*)-1-((4-bromophenyl)sulfonyl)-2-phenylaziridine **453a**



453a

Table 1 Crystal data and structure refinement for 453a.

Identification code	**453a**
Empirical formula	$C_{14}H_{12}NO_2SBr$
Formula weight	338.22
Temperature/K	120(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	5.4613(6)
b/Å	8.7109(8)
c/Å	28.546(3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1358.0(2)
Z	4
ρ_{calc}g/cm^3	1.654
μ/mm^{-1}	5.543
F(000)	680.0
Crystal size/mm^3	0.4767 × 0.2099 × 0.109
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.192 to 148.45
Index ranges	-6 ≤ h ≤ 6, -8 ≤ k ≤ 10, -28 ≤ l ≤ 35
Reflections collected	6714
Independent reflections	2661 [R_{int} = 0.0365, R_{sigma} = 0.0354]
Data/restraints/parameters	2661/0/172
Goodness-of-fit on F^2	1.070
Final R indexes [I>=2σ (I)]	R_1 = 0.0316, wR_2 = 0.0829
Final R indexes [all data]	R_1 = 0.0332, wR_2 = 0.0835
Largest diff. peak/hole / e Å^{-3}	0.33/-0.54
Flack parameter	-0.027(18)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 453a. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	-3013.1(16)	2989.0(11)	6055.0(3)	14.4(2)
N2	-1730(7)	4694(4)	6126.6(12)	16.6(7)
C3	732(7)	4856(5)	6339.1(13)	16.0(8)
C4	307(8)	5235(5)	5834.7(13)	19.8(8)
O5	-4633(5)	3149(3)	5662.6(9)	19.2(6)

O6	-3986(5)	2589(3)	6505(1)	20.6(6)
C7	-736(7)	1636(4)	5906.2(13)	15.9(8)
C8	515(8)	872(5)	6260.5(14)	20.3(9)
C9	2307(8)	-201(5)	6145.2(15)	21.0(9)
C10	2775(8)	-467(5)	5676.4(16)	20.6(9)
C11	1540(8)	280(5)	5318.6(15)	19.7(8)
C12	-238(8)	1350(4)	5436.4(13)	17.4(8)
Br13	5213.5(8)	-1937.0(5)	5512.0(2)	30.21(16)
C14	951(7)	6033(4)	6715.5(13)	16.1(8)
C15	-820(8)	7149(5)	6786.0(14)	23.0(9)
C16	-547(8)	8218(5)	7143.4(15)	25.2(9)
C17	1470(9)	8173(6)	7434.2(15)	27.5(10)
C18	3254(8)	7057(6)	7366.4(15)	26.2(9)
C19	3005(8)	5996(5)	7006.8(14)	22.1(9)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 453a. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	13.5(4)	13.3(4)	16.3(4)	-1.9(4)	0.9(3)	-0.3(4)
N2	17.2(17)	13.2(16)	19.4(16)	-2.0(13)	-1.1(13)	3.0(14)
C3	15.8(19)	13.7(18)	18.5(18)	1.6(15)	-1.2(15)	0.0(16)
C4	24(2)	16.2(18)	19.0(18)	-0.3(14)	1.1(17)	0.3(18)
O5	13.3(13)	22.8(14)	21.5(12)	-0.9(11)	-4.1(10)	2.5(13)
O6	21.5(15)	21.1(14)	19.2(13)	-0.1(12)	4.0(11)	-3.3(12)
C7	19(2)	9.3(18)	19.8(17)	0.1(14)	0.7(14)	-2.8(15)
C8	27(2)	14.1(18)	19.5(18)	-0.4(15)	0.4(17)	0.6(18)
C9	22(2)	11.2(18)	29(2)	2.3(16)	-6.3(17)	0.7(17)
C10	15(2)	13.6(19)	33(2)	-1.1(16)	3.7(16)	-1.2(16)
C11	22(2)	14.6(19)	22.6(19)	-3.7(16)	3.9(16)	-4.6(17)
C12	17.2(19)	13.4(17)	21.5(18)	1.6(14)	-0.4(16)	-2.6(16)
Br13	22.5(2)	19.4(2)	48.8(3)	-5.9(2)	2.76(19)	6.5(2)
C14	17.7(19)	12.1(19)	18.4(18)	0.3(15)	2.9(14)	0.0(16)
C15	24(2)	20(2)	25.0(19)	0.2(17)	1.4(15)	-1.2(18)
C16	27(2)	18(2)	31(2)	-6.7(18)	8.3(17)	3(2)
C17	35(2)	24(2)	23.0(19)	-10.3(19)	5.2(18)	-8(2)
C18	19(2)	34(3)	25(2)	-5(2)	0.3(15)	-6(2)
C19	21(2)	22(2)	23(2)	-2.0(17)	0.9(16)	-1.0(18)

Table 4 Bond Lengths for 453a.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	N2	1.655(3)	C9	C10	1.382(6)
S1	O5	1.434(3)	C10	C11	1.387(6)
S1	O6	1.433(3)	C10	Br13	1.906(4)
S1	C7	1.765(4)	C11	C12	1.387(6)
N2	C3	1.481(5)	C14	C15	1.386(6)
N2	C4	1.468(5)	C14	C19	1.397(6)
C3	C4	1.495(5)	C15	C16	1.389(6)
C3	C14	1.490(5)	C16	C17	1.380(7)
C7	C8	1.390(5)	C17	C18	1.390(7)
C7	C12	1.391(5)	C18	C19	1.388(6)
C8	C9	1.393(6)			

Table 5 Bond Angles for 453a.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
N2	S1	C7	109.30(19)	C7	C8	C9	119.6(4)
O5	S1	N2	105.71(17)	C10	C9	C8	118.1(4)
O5	S1	C7	108.14(17)	C9	C10	C11	123.0(4)
O6	S1	N2	105.32(17)	C9	C10	Br13	118.7(3)
O6	S1	O5	119.68(17)	C11	C10	Br13	118.3(3)
O6	S1	C7	108.34(18)	C10	C11	C12	118.5(4)
C3	N2	S1	121.3(3)	C11	C12	C7	119.4(4)
C4	N2	S1	122.6(3)	C15	C14	C3	122.1(4)
C4	N2	C3	60.9(3)	C15	C14	C19	119.3(4)
N2	C3	C4	59.1(3)	C19	C14	C3	118.6(4)
N2	C3	C14	115.7(3)	C14	C15	C16	120.1(4)
C14	C3	C4	123.7(4)	C17	C16	C15	120.6(4)
N2	C4	C3	60.0(2)	C16	C17	C18	119.7(4)
C8	C7	S1	119.4(3)	C19	C18	C17	120.0(4)
C8	C7	C12	121.3(4)	C18	C19	C14	120.3(4)
C12	C7	S1	119.3(3)				

Table 6 Torsion Angles for 453a.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
S1	N2	C3	C4	112.5(3)	O6	S1	C7	C8	-26.4(4)
S1	N2	C3	C14	-132.0(3)	O6	S1	C7	C12	153.3(3)
S1	N2	C4	C3	-110.5(3)	C7	S1	N2	C3	-36.4(3)
S1	C7	C8	C9	179.8(3)	C7	S1	N2	C4	37.1(4)
S1	C7	C12	C11	-179.6(3)	C7	C8	C9	C10	-0.1(6)
N2	S1	C7	C8	87.9(4)	C8	C7	C12	C11	0.1(6)
N2	S1	C7	C12	-92.5(4)	C8	C9	C10	C11	-0.1(6)
N2	C3	C14	C15	-15.0(5)	C8	C9	C10	Br13	-179.8(3)
N2	C3	C14	C19	164.6(4)	C9	C10	C11	C12	0.3(6)
C3	C14	C15	C16	179.6(4)	C10	C11	C12	C7	-0.3(6)
C3	C14	C19	C18	-178.9(4)	C12	C7	C8	C9	0.1(6)

C4	N2	C3	C14	115.4(4)	Br13	C10	C11	C12	180.0(3)
C4	C3	C14	C15	53.6(6)	C14	C3	C4	N2	-102.0(4)
C4	C3	C14	C19	-126.8(4)	C14	C15	C16	C17	-0.6(6)
O5	S1	N2	C3	-152.5(3)	C15	C14	C19	C18	0.7(6)
O5	S1	N2	C4	-79.1(3)	C15	C16	C17	C18	0.6(7)
O5	S1	C7	C8	-157.5(3)	C16	C17	C18	C19	0.1(7)
O5	S1	C7	C12	22.2(4)	C17	C18	C19	C14	-0.7(7)
O6	S1	N2	C3	79.9(3)	C19	C14	C15	C16	0.0(6)
O6	S1	N2	C4	153.3(3)					

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 453a.

Atom	*x*	*y*	*z*	U(eq)
H3	1620	3871	6399	19
H4A	323	6331	5743	24
H4B	971	4521	5596	24
H8	148	1081	6580	24
H9	3184	-734	6382	25
H11	1903	65	5000	24
H12	-1108	1883	5198	21
H15	-2223	7183	6590	28
H16	-1759	8986	7188	30
H17	1638	8901	7680	33
H18	4645	7021	7566	31
H19	4236	5242	6959	26

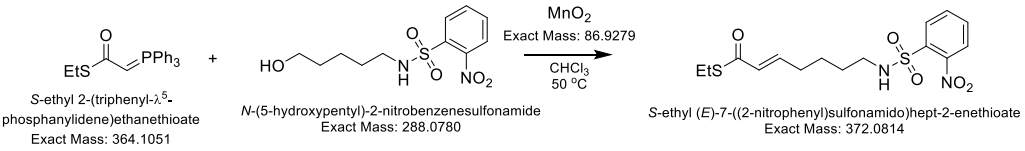
[2] Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.

[3] Bourhis, L.J., Dolomanov, O.V., Gildea, R.J., Howard, J.A.K., Puschmann, H. (2015). Acta Cryst. A71, 59-75.

[4] Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122

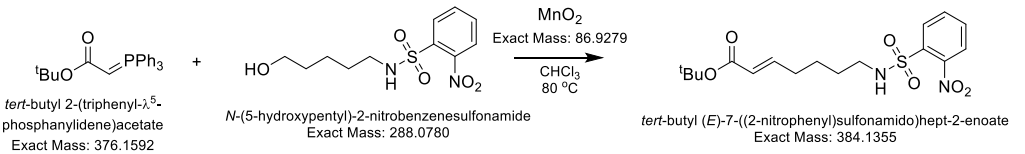
Example of typical synthesis

Typical synthesis of S-ethyl (E)-7-((2-nitrophenyl)sulfonamido)hept-2-enethioate



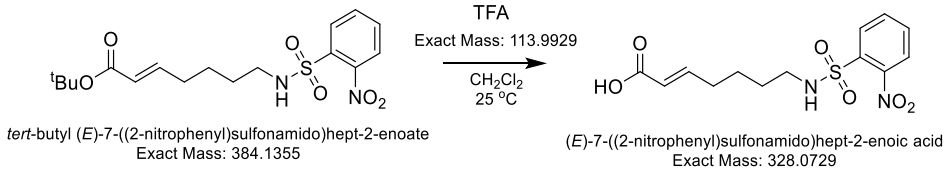
A suspension of manganese dioxide (72.4 g, 832 mmol, 8 equiv.) in a solution of S-ethyl 2-(triphenyl-l5-phosphanylidene)ethanethioate (41.7 g, 114.6 mmol, 1.1 equiv.) and *N*-(5-hydroxypentyl)-2-nitrobenzenesulfonamide (30 g, 104 mmol, 1 equiv.) in chloroform (0.1 or 0.2M) is heated at 50 °C for approximately 2 days until the reaction is seen to progress to completion by ^{1}H NMR spectroscopy. The reaction is then passed through a celite pad with chloroform (typically 2-3 times reaction solvent volume or until celite pad is colourless) and the resulting solution is concentrated under reduced pressure. The product is then purified by flash column chromatography on silica gel (4:1, petroleum ether/ethyl acetate, *Rf*=0.17).

Typical synthesis of *tert*-butyl (*E*)-7-((2-nitrophenyl)sulfonamido)hept-2-enoate



A suspension of manganese dioxide (72.4 g, 832 mmol, 8 equiv.) in a solution of *tert*-butyl 2-(triphenyl-l5-phosphanylidene)acetate (43.1 g, 114.6 mmol, 1.1 equiv.) and *N*-(5-hydroxypentyl)-2-nitrobenzenesulfonamide (30 g, 104 mmol, 1 equiv.) in chloroform (0.1 or 0.2M) is heated at 80 °C for approximately 2 days until the reaction is seen to progress to completion by ^{1}H NMR spectroscopy. The reaction is then passed through a celite pad with chloroform (typically 2-3 times reaction solvent volume or until celite pad is colourless) and the resulting solution is concentrated under reduced pressure. The product is then purified by flash column chromatography on silica gel (4:1, petroleum ether/ethyl acetate, *Rf*=0.17).

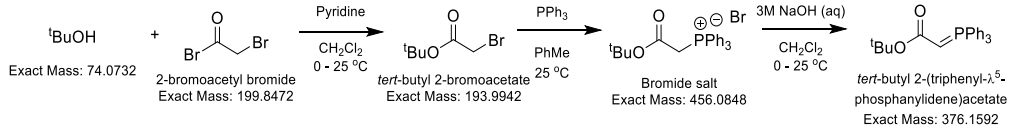
Typical synthesis of (*E*)-7-((2-nitrophenyl)sulfonamido)hept-2-enoic acid



A solution of *tert*-butyl (*E*)-7-((2-nitrophenyl)sulfonamido)hept-2-enoate (20 g, 52 mmol, 11 equiv.) and trifluoroacetic acid (59 g, 39.8 mL, 521 mmol, 10 equiv.) in dichloromethane (20 mL, typically one third the volume of TFA) is

stirred at room temperature for 16 h until the ester is no longer present by TLC or ^{1}H NMR, the solution is then concentrated under reduced pressure and the residue is passed through a small silica plug to remove any colour with neat ethyl acetate. The resulting solution is then concentrated under reduced pressure and recrystallized from ethyl acetate/petroleum ether to give the acid as a colourless solid.

Typical synthesis of *tert*-butyl 2-(triphenyl-l5-phosphanylidene)acetate



To a solution of *tert*-butanol (14.8 g, 200 mmol, 1 equiv.) and pyridine (16 mL, 200 mmol, 1 equiv.) in dichloromethane (150 mL) is added dropwise by a pressure equalising addition funnel bromoacetyl bromide (40 g, 17.2 mL, 200 mmol, 1 equiv.) in dichloromethane (50 mL) over a period of thirty minutes. The solution is then allowed to stir for 2 h at which point brine (150 mL) is added. The organic phase is then further washed with 2M hydrochloric acid (2 x 100 mL), dried over magnesium sulfate and concentrated under reduced pressure to give the product which is used crude in the next step (typically ~90%).

To the crude *tert*-butyl 2-bromoacetate (34.9 g, 180 mmol, 1 equiv.) is dissolved in toluene (200 mL) and triphenyl phosphine (47 g, 180 mmol, 1 equiv.) in toluene (200 mL) and the solution is stirred at a high rate (1000-1200 RPM) for 16 h (The high rate of stirring seems to help the resulting precipitate crash out as a powder as opposed to an oil). The precipitate is then filtered off and washed with toluene (100 mL) and diethyl ether (2 x 150 mL) to give the bromide salt as a colourless powder (typically ~85%).

The bromide salt (69.7 g, 153 mmol, 1 equiv.) is then dissolved in dichloromethane (200 mL) and 3M sodium hydroxide solution is added (~50 mL), the organic layer normally becomes a dark red colour, the biphasic mixture is then stirred for 2 h before being transferred to a separating funnel and the organic is washed with brine (2 x 150 mL), dried over magnesium sulfate and concentrated under reduced pressure. The resulting beige/brown solid is then used without any further purification, it can be recrystallized somewhat from ethyl acetate/ petroleum ether but this makes little difference in future reactions.

End Of Appendix