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Synthesis, crystal structures and anti-inflammatory activity of four 3,5-bis(arylidene)-*N*-benzenesulfonyl-4-piperidone derivatives

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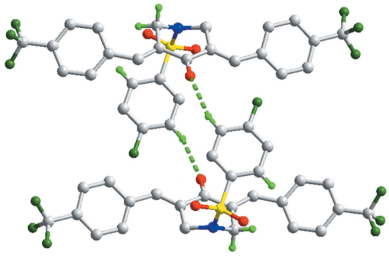
3,5-Bis(arylidene)-4-piperidone (BAP) derivatives display good antitumour and anti-inflammatory activities because of their double α,β-unsaturated ketone structural characteristics. If *N*-benzenesulfonyl substituents are introduced into BAPs, the configuration of the BAPs would change significantly and their anti-inflammatory activities should improve. Four *N*-benzenesulfonyl BAPs, namely (3*E*,5*E*)-1-(4-methylbenzenesulfonyl)-3,5-bis[4-(trifluoromethyl)benzylidene]-piperidin-4-one dichloromethane monosolvate, $C_{28}H_{21}F_6NO_3S\cdot CH_2Cl_2$, (**4**), (3*E*,5*E*)-1-(4-fluorobenzenesulfonyl)-3,5-bis[4-(trifluoromethyl)benzylidene]-piperidin-4-one, $C_{27}H_{18}F_7NO_3S$, (**5**), (3*E*,5*E*)-1-(4-nitrobenzenesulfonyl)-3,5-bis[4-(trifluoromethyl)benzylidene]piperidin-4-one, $C_{27}H_{18}F_6N_2O_5S$, (**6**), and (3*E*,5*E*)-1-(4-cyanobenzenesulfonyl)-3,5-bis[4-(trifluoromethyl)benzylidene]piperidin-4-one dichloromethane monosolvate, $C_{28}H_{18}F_6N_2O_3S\cdot CH_2Cl_2$, (**7**), were prepared by Claisen–Schmidt condensation and *N*-sulfonylation. They were characterized by NMR, FT–IR and HRMS (high resolution mass spectrometry). Single-crystal structure analysis reveals that the two 4-(trifluoromethyl)phenyl rings on both sides of the piperidone ring in (**4**)–(**7**) adopt an *E* stereochemistry of the olefinic double bonds. Molecules of both (**4**) and (**6**) are connected by hydrogen bonds into one-dimensional chains. In (**5**) and (**7**), pairs of adjacent molecules embrace through intermolecular hydrogen bonds to form a bimolecular combination, which are further extended into a two-dimensional sheet. The anti-inflammatory activity data reveal that (**4**)–(**7**) significantly inhibit LPS-induced interleukin (IL-6) and tumour necrosis factor (TNF-α) secretion. Most importantly, (**6**) and (**7**), with strong electron-withdrawing substituents, display more potential inhibitory effects than (**4**) and (**5**).

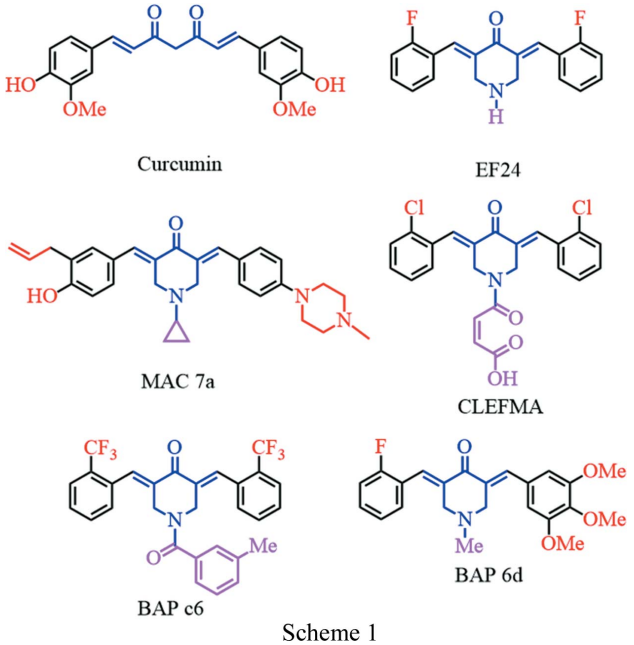
1. Introduction

Inflammation is an immune response to injury, tissue ischemia, infectious agents and many pathological processes of chronic diseases, especially, the development of tumours (Mantovani *et al.*, 2008; Medzhitov, 2010; Nieminen *et al.*, 2010; Shin *et al.*, 2014). Epidemiological studies have shown that individuals with ulcerative colitis may have a tenfold greater likelihood of developing colorectal carcinoma (Hagemann *et al.*, 2007). Also, hepatocellular carcinoma (HCC) could slowly unfold from chronic inflammation mainly triggered by exposure to infectious agents or to toxic compounds (Berasain *et al.*, 2009; El-Serag & Rudolph, 2007). Therefore, anti-inflammation plays a key role in the prevention of the development of tumours.

Macrophages, an innate immune cell, can drive tumour progression through the production of pro-inflammatory mediators, such as interleukin 6 (IL-6) and tumour necrosis factor-α (TNF-α), which are involved in the pathogenesis of



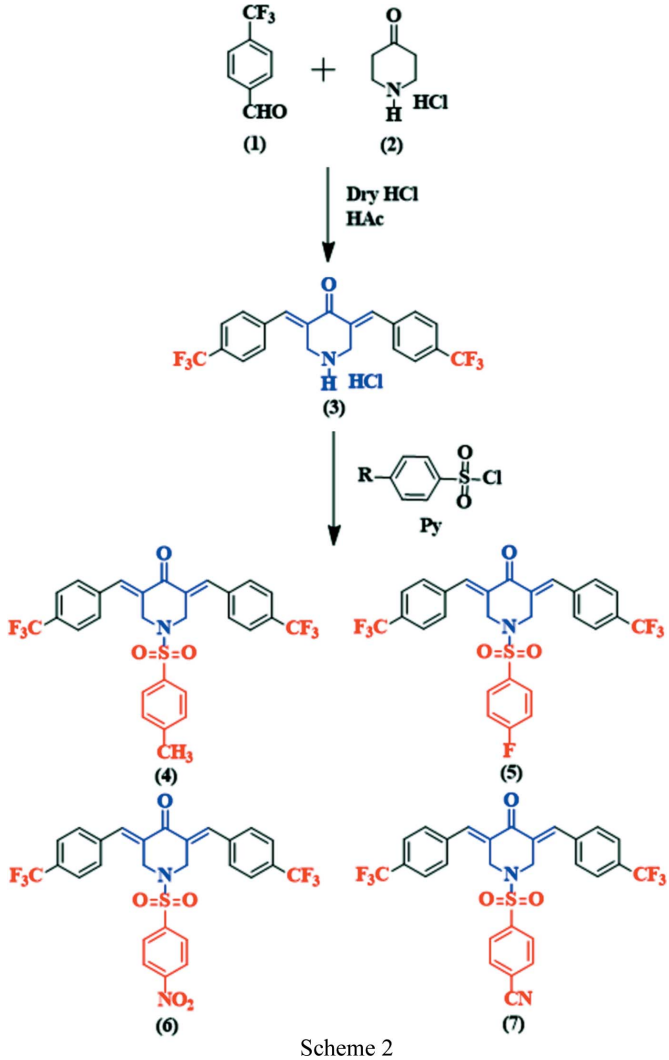
tumours (Greten *et al.*, 2004). Moreover, the expression of the inflammatory cytokines, including IL-6 and TNF-α, can be down-regulated by inhibiting the activation of free-radical-activated transcription factors, such as transcription factor nuclear factor-κB (NF-κB), which provides a critical link between inflammation and tumour (Karin, 2009). It has been observed that NF-κB activation is a result of underlying inflammation or a consequence of the formation of an inflammatory microenvironment during tumour progression (Chen *et al.*, 2017). These observations suggest that NF-κB is a potential target for the design of new anticancer drugs with anti-inflammatory activity.



Scheme 1

Curcumin [1,7-bis(4-hydroxy-3-methoxyphenyl)hepta-1,6-dien-3,5-dione, see Scheme 1], a natural component of the rhizome of *Curcuma longa*, has been proved to be a powerful chemopreventive and antitumour agent with anti-inflammatory, antibacterial and antioxidant properties. However, the clinical use of curcumin has been limited because of its low anticancer activity and poor bioavailability (Nelson *et al.*, 2017; Prakobwong *et al.*, 2011). In order to overcome these defects, many improved curcumin analogues have been reported. 3,5-Bis(arylidene)-4-piperidone (BAP) derivatives are analogues which display good antitumour and anti-inflammatory activities. In BAPs, the 1,5-diaryl-3-oxopenta-1,4-dienyl pharmacophore, with two α,β-unsaturated ketone groups, shows significant preference or sequential affinity for biothiols compared to amino and hydroxy groups, which results in an improved chemosensitivity to tumour cells over normal cells (Lagisetty *et al.*, 2010; Das *et al.*, 2010; Sun *et al.*, 2016; Li *et al.*, 2017, 2018).

3,5-Bis(2-fluorobenzylidene)-4-piperidone (EF24, Scheme 1) is an effective antitumour agent that inhibits tumour growth and metastasis by inhibiting NF-κB-dependent signalling pathways (Yin *et al.*, 2016) or IKKβ kinase activity (Kasinski *et al.*, 2008), inducing G2/M arrest and apoptosis (Selvendiran *et al.*, 2007). CLEFMA (Scheme 1) can induce apoptosis by cleavage of caspases 3/9 and PARP (Yadav *et al.*, 2013). Another alkylated monocarbonyl analogue of curcumin (MAC 7a, Scheme 1) showed potent anti-inflammatory activity against lipopolysaccharide (LPS)-induced acute lung injury in rats by inhibiting the expression of IL-6, IL-1β, TNF-α and vascular cell adhesion molecule (VCAM-1) in Beas-2B cells (Zhu *et al.*, 2016). *N*-Benzoyl BAP c6 (Scheme 1) displayed potential anti-inflammatory activity by inhibiting LPS-stimulated TNF-α, IL-6, IL-1β and nitric oxide production in RAW 264.7 cells (Xie *et al.*, 2017). Taken together, BAPs possess excellent antitumour and anti-inflammatory activities. More importantly, BAPs can be structurally modified readily to potentially improve bioactivity, whereas cucurmin is difficult to modify.



Scheme 2

Recently, we have reported many novel symmetric and unsymmetric BAPs (such as BAP 6d, see Scheme 1) as antitumour agents (Li *et al.*, 2015, 2018; Sun *et al.*, 2016; Chen *et al.*, 2016; Liu *et al.*, 2016; Yao *et al.*, 2018). We hypothesize that by changing the *N*-methyl substituent into *N*-benzenesulfonyl substituents in BAPs, the configuration of the BAPs would change markedly and this might improve the antitumour and anti-inflammatory activities of these compounds. In this study, four 3,5-bis(arylidene)-*N*-benzenesulfonyl-4-piperidone deri-

Table 1
Experimental details.

	(**4**)	(**5**)	(**6**)	(**7**)
Crystal data				
Chemical formula	$C_{28}H_{21}F_6NO_3S{\cdot}CH_2Cl_2$	$C_{27}H_{18}F_7NO_3S$	$C_{27}H_{18}F_6N_2O_5S$	$C_{28}H_{18}F_6N_2O_3S{\cdot}CH_2Cl_2$
M_r	650.44	569.48	596.49	661.43
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	150	150	150	150
a, b, c (Å)	8.6718 (5), 23.5083 (12), 14.3939 (9)	8.5664 (6), 10.0932 (8), 14.3871 (12)	7.4346 (7), 11.6714 (11), 14.6479 (15)	9.7338 (6), 12.4558 (9), 12.6397 (9)
α, β, γ (°)	90, 102.962 (2), 90	90.590 (4), 98.592 (4), 97.309 (4)	90.825 (5), 96.884 (5), 98.971 (5)	70.586 (3), 88.379 (4), 81.726 (3)
V (Å^3)	2859.6 (3)	1219.44 (17)	1245.7 (2)	1429.97 (17)
Z	4	2	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.37	0.22	0.22	0.38
Crystal size (mm)	0.14 × 0.10 × 0.10	0.26 × 0.17 × 0.10	0.18 × 0.16 × 0.10	0.20 × 0.15 × 0.11
Data collection				
Diffractometer	Bruker SMART CCD area detector	Bruker SMART CCD area detector	Bruker SMART CCD area detector	Bruker SMART CCD area detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	38920, 5852, 4236	21256, 4790, 3187	23092, 4848, 3350	24790, 5604, 3869
R_{int}	0.062	0.074	0.072	0.070
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.626	0.617	0.617	0.617
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.126, 1.04	0.050, 0.129, 1.01	0.051, 0.129, 1.03	0.046, 0.109, 1.01
No. of reflections	5852	4790	4848	5604
No. of parameters	408	380	370	388
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.69, −0.65	0.48, −0.38	0.41, −0.41	0.53, −0.44

Computer programs: *APEX2* (Bruker, 2007), *SAINT* (Bruker, 2012), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *SHELXTL* (Sheldrick, 2008).

vatives, *i.e.* (**4**)–(**7**), were synthesized (Scheme 2) and their structures confirmed by single-crystal X-ray diffraction.

2. Experimental

2.1. Chemistry

4-(Trifluoromethyl)benzaldehyde, (**1**), 4-piperidone hydrate hydrochloride, (**2**), 4-fluorobenzenesulfonyl chloride, 4-nitrobenzenesulfonyl chloride and 4-cyanobenzenesulfonyl chloride were purchased from Sinopharm Chemical Reagent Co. Ltd (Shanghai, China). ^{1}H NMR data were collected using a Bruker Avance 400 MHz spectrometer. ^{13}C NMR data were collected at 100 MHz on a Bruker Avance 400 MHz spectrometer or at 150 MHz on a Bruker Avance 600 MHz spectrometer. Chemical shifts were reported in δ relative to TMS (tetramethylsilane). HRMS (FTMS) were performed on a Finnigan-MAT-95 mass spectrometer. IR spectra were obtained in the 400–4000 cm^{-1} range using a PerkinElmer Frontier Mid-IR FT–IR spectrometer.

2.2. Synthesis and crystallization of (4)–(7)

4-(Trifluoromethyl)benzaldehyde, (**1**) (1.74 g, 10.0 mmol), and 4-piperidone hydrate hydrochloride, (**2**) (0.77 g, 5.0 mmol), were dissolved in acetic acid (10 ml). Dry hydrogen chloride gas was passed through this mixture for 35 min and a clear solution was obtained. After stirring at room temperature (RT) for about 24 h [monitored by thin-layer chromatography (TLC)], the precipitates were collected and washed with acetone to obtain intermediate (**3**). (**3**) (0.45 g, 1.0 mmol) and benzenesulfonyl chloride [4-toluenesulfonyl chloride (0.21 g, 1.1 mmol), 4-fluorobenzenesulfonyl chloride (0.21 g, 1.1 mmol), 4-nitrobenzenesulfonyl chloride (0.24 g, 1.1 mmol) or 4-cyanobenzenesulfonyl chloride (0.22 g, 1.1 mmol) for (**4**)–(**7**), respectively] were dissolved in dichloromethane (20 ml). Two drops of pyridine were added to the mixture, which was then stirred for about 6 h at RT (monitored by TLC). The precipitates were collected, washed with water and recrystallized from dichloromethane/petroleum ether (1:1 *v*/*v*) as light-yellow powders of (**4**)–(**7**). Single crystals were prepared by slow evaporation of dichloromethane/methanol solutions of (**4**)–(**7**) at RT.

2.2.1. Analytical data for (4). Light-yellow powder; yield: 81%; m.p. 465∼467 K. IR (cm^{-1}): 1674 (*m*), 1614 (*s*), 1394 (*m*), 1325 (*s*), 1261 (*m*), 1243 (*s*), 1161 (*s*), 1088 (*m*), 1069 (*s*), 1037 (*m*), 1016 (*s*), 986 (*s*), 840 (*s*), 804 (*m*), 765 (*m*), 738 (*s*), 694 (*s*), 672 (*s*). ^{1}H NMR (600 MHz, DMSO-d_6): δ 7.88 (*d*, *J* = 8.0 Hz, 4H), 7.71 (*d*, *J* = 7.9 Hz, 4H), 7.65 (*s*, 2H), 7.44 (*d*, *J* = 7.9 Hz, 2H), 7.37 (*d*, *J* = 7.9 Hz, 2H), 4.64 (*s*, 4H), 2.41 (*s*, 3H). ^{13}C NMR (150 MHz, DMSO-d_6): δ 21.0, 46.5, 124.0 (*q*, *J* = 270.6 Hz, CF_3), 125.6 (*q*, *J* = 5.6 Hz, 2C-CF_3), 127.2, 129.3 (*q*, *J* = 32.2 Hz, 1C-CF_3), 129.9, 130.9, 132.5, 134.2, 135.6, 137.9, 144.1, 184.0. HRMS (FTMS) calculated for $C_{28}H_{22}F_6NO_3S$ [M + H$^+$] 566.1225, found 566.1229.

2.2.2. Analytical data for (5). Light-yellow powder; yield: 83%; m.p. 515–517 K. IR (cm^{-1}): 1674 (*s*), 1615 (*s*), 1585 (*m*), 1529 (*s*), 1371 (*m*), 1321 (*s*), 1261 (*m*), 1238 (*m*), 1169 (*s*), 1113 (*s*), 1069 (*s*), 1041 (*m*), 1015 (*m*), 992 (*m*), 968 (*m*), 937 (*m*), 848 (*s*), 827 (*s*), 770 (*m*), 735 (*s*), 698 (*m*). ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.89 (*d*, *J* = 8.3 Hz, 4H), 7.73 (*d*, *J* = 8.1 Hz, 4H), 7.64 (*s*, 2H), 7.59 (*m*, 2H), 7.41 (*m*, 2H), 4.70 (*s*, 4H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 47.3, 117.4 (*d*, $^2J_{CF}$ = 22.4 Hz), 124.7 (*q*, *J* = 271.4 Hz, CF_3), 126.4 (*q*, *J* = 3.2 Hz, 2C-CF_3), 130.1 (*q*, *J* = 32.6 Hz, 1C-CF_3), 131.2 (*d*, $^3J_{CF}$ = 9.6 Hz), 131.8, 133.0, 134.5 (*d*, $^4J_{CF}$ = 2.8 Hz), 136.5, 138.6, 165.5 (*d*, $^1J_{CF}$ = 251.2 Hz), 184.7. HRMS (FTMS) calculated for $C_{27}H_{19}F_7NO_3S$ [*M* + H^+] 570.0974, found 570.0972.

2.2.3. Analytical data for (6). Light-yellow powder; yield: 86%; m.p. 518–520 K. IR (cm^{-1}): 1673 (*s*), 1613 (*s*), 1589 (*s*), 1327 (*s*), 1242 (*s*), 1188 (*m*), 1163 (*s*), 1113 (*s*), 1086 (*m*), 1071 (*s*), 1038 (*s*), 1016 (*s*), 986 (*s*), 942 (*m*), 847 (*s*), 818 (*m*), 803 (*s*), 738 (*s*). ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.38 (*d*, *J* = 9.2 Hz, 2H), 7.89 (*d*, *J* = 8.3 Hz, 4H), 7.81 (*d*, *J* = 9.2 Hz, 2H), 7.74 (*d*, *J* = 8.2 Hz, 4H), 7.65 (*s*, 2H), 4.75 (*s*, 4H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 47.3, 124.7 (*q*, *J* = 270.7 Hz, CF_3), 125.5, 126.4 (*q*, *J* = 3.7 Hz, C-CF_3), 129.6, 130.2 (*q*, *J* = 31.7 Hz, C-CF_3), 131.8, 132.7, 136.9, 138.5, 143.6, 150.8, 184.7. HRMS (FTMS) calculated for $C_{27}H_{19}F_6N_2O_5S$ [*M* + H^+] 597.0919, found 597.0923.

2.2.4. Analytical data for (7). Light-yellow powder; yield: 81%; m.p. 496–498 K. IR (cm^{-1}): 2237 (*s*), 1675 (*s*), 1614 (*s*), 1414 (*m*), 1369 (*s*), 1322 (*s*), 1266 (*m*), 1239 (*m*), 1169 (*s*), 1113 (*m*), 1068 (*s*), 1042 (*s*), 1016 (*s*), 992 (*s*), 936 (*s*), 829 (*s*), 742 (*m*), 699 (*s*). ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.07 (*m*, 2H), 7.89 (*d*, *J* = 8.4 Hz, 4H), 7.73 (*d*, *J* = 8.4 Hz, 4H), 7.68 (*m*, 2H), 7.62 (*s*, 2H), 4.74 (*s*, 4H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 47.3, 116.6, 118.3, 124.7 (*q*, *J* = 271.2 Hz, CF_3), 126.4 (*q*, *J* = 3.7 Hz, 2C-CF_3), 128.7, 130.1 (*q*, *J* = 31.8 Hz, 1C-CF_3), 131.8, 132.7, 134.3, 136.7, 138.5, 142.3, 184.7. HRMS (FTMS) calculated for $C_{28}H_{19}F_6N_2O_3S$ [*M* + H^+] 577.1021, found 577.1024.

2.3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The methylene H atoms of (**4**)–(**7**) and the methyl H atoms of (**4**) were placed in geometrically idealized positions and constrained to ride on their parent C atoms, with C—H = 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene, and C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms. In (**4**), the trifluoromethyl groups were disordered, with refined site-occupation factors of 0.51 (3) and 0.49 (3) for atoms F4/F5/F6 and F4′/F5′/F6′, respectively. In (**5**), the trifluoromethyl groups were disordered, with refined site-occupation factors of 0.532 (19) and 0.458 (19) for atoms F1/F2/F3 and F1′/F2′/F3′, respectively. Geometric restraints in (**4**) and (**5**) were used for modelling the trifluoromethyl-group disorder.

2.4. Anti-inflammatory evaluation of (4)–(7)

The anti-inflammatory effects of compounds (**4**)–(**7**) were evaluated by inhibition of TNF-α and IL-6 secretion from RAW264.7 macrophages stimulated by LPS using an ELISA kit (eBioScience, CA, USA). Briefly, RAW264.7 cells were pretreated with 30 µ*M* of pyrrolidinedithiocarbamate (PDTC) or with 10 µ*M* of compounds for 2 h. After treatment with LPS (1 µg ml^{-1}) for 22 h, the culture media were centrifuged and collected. The optical density was determined using a Thermo Multiskan FC at 450 nm (Thermo Scientific, MA, USA).

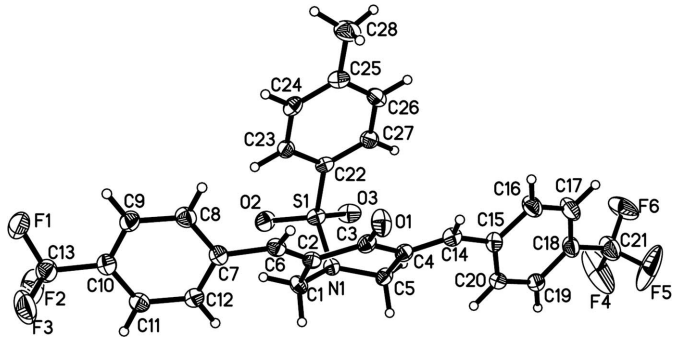


Figure 1
A perspective view of (**4**), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Disordered atoms F4′, F5′ and F6′ in the trifluoromethyl group have been omitted for clarity, as has the dichloromethane solvent molecule.

3. Results and discussion

3.1. Synthetic analysis

The title compounds (**4**)–(**7**) were synthesized using standard protocols for Claisen–Schmidt condensation and *N*-sulfonylation. The synthetic strategy and structures of (**4**)–(**7**) are shown in Scheme 2. The Claisen–Schmidt condensation reaction was catalyzed by dry HCl. The harvested intermediate (**3**) was stable at room temperature. The *N*-sulfonylation reaction was catalyzed by pyridine. The target compounds were filtered off, washed with water and recrystallized from dichloromethane/petroleum ether. The overall yields of (**4**)–(**7**) were more than 80%, because there were symmetric products as opposed to dissymmetric BAPs (Li *et al.*, 2018).

3.2. Structural characterization

The structures of (**4**)–(**7**) were fully characterized by ^{1}H NMR, ^{13}C NMR, FT–IR and HRMS (high resolution mass

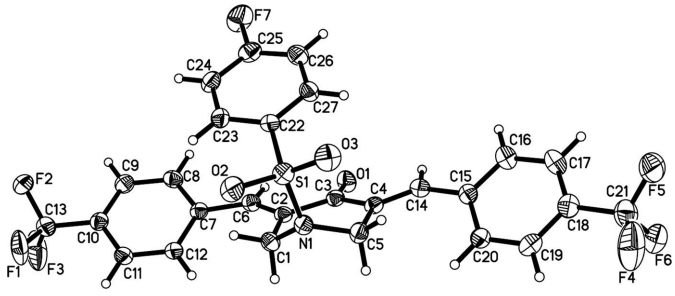


Figure 2
A perspective view of (**5**), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Disordered atoms F1′, F2′ and F3′ in the trifluoromethyl group have been omitted for clarity.

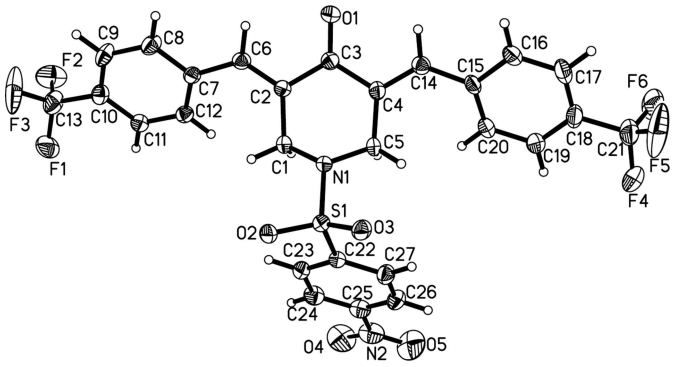


Figure 3
A perspective view of (**6**), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

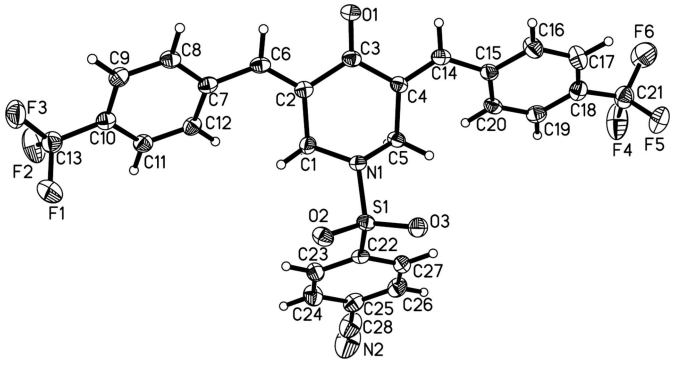


Figure 4
A perspective view of (**7**), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The dichloromethane solvent molecule has been omitted for clarity.

spectrometry). Based on the ^{1}H NMR data, there is only an evident unimodal signal in the range 7.65∼7.62 ppm, corresponding to the proton signals of –C═CH– in the α,β-unsaturated ketone. Meanwhile, there is only a distinct unimodal signal in the range 4.75∼4.64 ppm, corresponding to the proton signals of the $-CH_2$ groups of the central piperidone ring. Based on the ^{13}C NMR data, a carbonyl C atom can be found around 184 ppm.

From the FT–IR spectral data, the characteristic band at 1673∼1675 cm^{-1} is attributed to the stretching vibration of the C═O group of piperidone in (**4**)–(**7**). The strong bands at 1615∼1613 cm^{-1} are due to the stretching vibration of the C═C group in the α,β-unsaturated ketone. The strong bands at 1169∼1161 cm^{-1} are attributed to the sulfonamide group ($-SO_2N-$). In addition, the presence of a cyano group (CN) in (**7**) can be confirmed by the characteristic bands at around 2237 cm^{-1} (stretching vibration of –CN). Additionally, HRMS further confirmed the molecular weights and are consistent with the calculated results.

3.3. Crystal structure analysis

Single-crystal structure analysis shows that (**4**) crystallizes in the monoclinic space group $P2_1/n$, while (**5**)–(**7**) crystallize in the triclinic space group $P\overline{1}$. The asymmetric units of (**4**)–(**7**) contain one drug molecule each (Figs. 1–4). The main framework consists of 3,5-bis(arylidene)-*N*-benzenesulfonyl-4-piperidone with different substituents ($-CH_3$, –F, $-NO_2$ and –CN) on the *N*-benzenelfonyl group, resulting in significantly different molecular configurations.

The four molecules (**4**)–(**7**) are similar in that both sides of the 3,5-bis(arylidene)-4-piperidone ring expand in a linear fashion, with the two 4-(trifluoromethyl)phenyl rings adopting an *E* stereochemistry of the olefinic double bonds and thus the *E*,*E* isomer (Li *et al.*, 2018; Sun *et al.*, 2016). In addition, the values of the θ(C2—C6—C7—C12) and θ(C4—C14—C15—C20) torsion angles show similar structural features, which may assist in gleaning further knowledge of the structural features influencing cytotoxicity (Dimmock *et al.*, 2002). The θ(C2—C6—C7—C12) and θ(C4—C14—C15—C20) torsion angles are −32.9 (4) and 29.5 (4)° in (**4**), −30.2 (5) and 30.7 (4)° in (**5**), 30.2 (4) and −27.5 (5)° in (**6**), and −28.6 (4) and 32.8 (4)° in (**7**), respectively. The dihedral angle between the two 4-(trifluoromethyl)phenyl rings is 24.1 (3)° in (**4**),

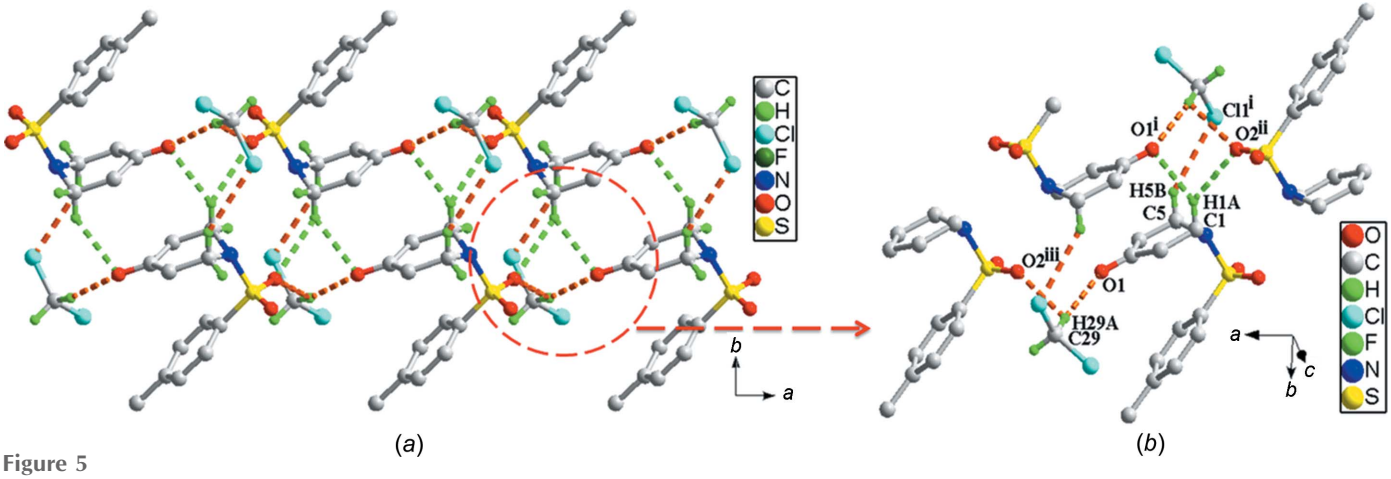


Figure 5
(*a*) The one-dimensional chain formed through intermolecular hydrogen bonds in (**4**). (*b*) The detailed hydrogen bonding, indicated by dashed lines. The 4-(trifluoromethyl)benzylidene groups have been omitted for clarity. [Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x, -y, -z$; (iii) $x+1, y, z$.]

Table 2
Hydrogen-bond geometry (Å, °) for (**4**).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O1^{i}	0.99	2.53	3.390 (3)	145
C1—H1A···O2^{ii}	0.99	2.61	3.365 (3)	133
C5—H5B···Cl1^{i}	0.99	2.98	3.832 (2)	144
C29—H29A···O1	0.99	2.34	3.279 (3)	159
C29—H29A···O2^{iii}	0.99	2.60	3.162 (3)	116

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x, -y, -z$; (iii) $x+1, y, z$.

18.6 (2)° in (**5**), 7.0 (2)° in (**6**) and 15.0 (2)° in (**7**), the dihedral angle in (**6**) being slightly smaller than the others.

The four structures differ markedly in the conformation of the *N*-arylsulfonyl groups. In (**4**) and (**5**), the arylsulfonyl group adopts a pseudo-axial conformation with respect to the 4-pyridone ring, with C2—C1—N1—S1 = −88.3 (2)° in (**4**) and 89.4 (2)° in (**5**), with the arylsulfonyl ring *endo* with respect to the 4-pyridone ring. In contrast, in (**6**) and (**7**), the arylsulfonyl group adopts a pseudo-equatorial conformation, with C2—C1—N1—S1 = 154.2 (2)° in (**6**) and 149.8 (2)° in (**7**). As shown in Figs. 1 and 2, (**4**) or (**5**) look like an 'organic clip' (Hou *et al.*, 2012). This could be caused by the different substituents of the *N*-benzenesulfonyl group and the packing.

As shown in Fig. 5, the molecular units of (**4**) are connected by intermolecular C—H···O and C—H···Cl hydrogen bonds into a one-dimensional antiparallel double chain which extends along the crystallographic *a* axis (Fig. 5*a*). Relevant hydrogen-bonding geometries are given in Table 2. As a hydrogen-bonding donor and acceptor, the dichloromethane solvent molecule plays an important role in the formation of the chain and links three BAP molecules through three hydrogen bonds (Table 2 and Fig. 5*b*).

As shown in Fig. 6(*a*), adjacent BAP molecules in (**5**) are joined through intermolecular C26—H26···O1^{i} hydrogen bonds to generate a bimolecular macrocycle, from which four 4-(trifluoromethyl)benzylidene groups extend. Based on the

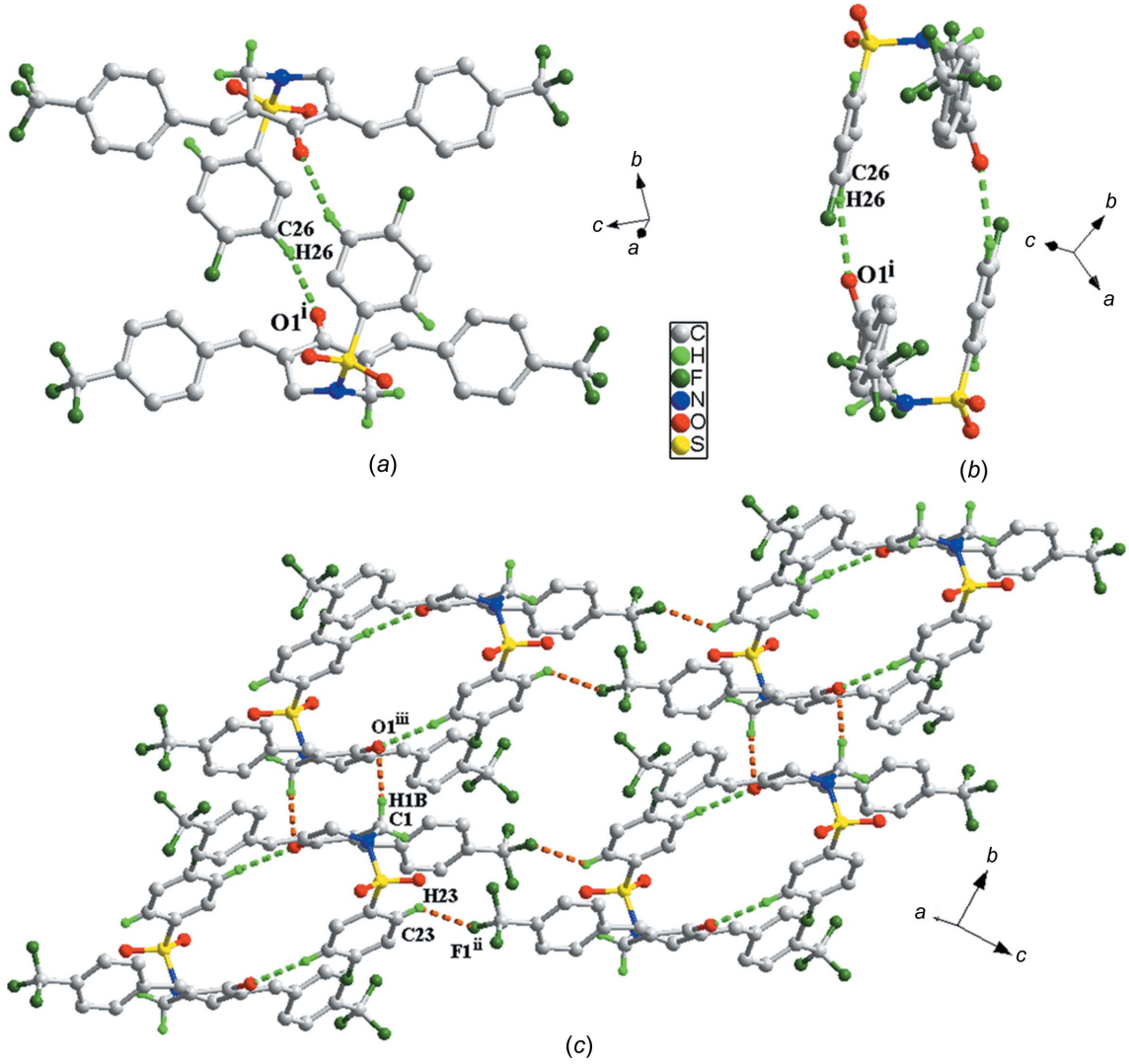


Figure 6
(*a*) The bimolecular macrocycle formed through intermolecular C26–H26···O1^{i} hydrogen bonds in (**5**). (*b*) A top view of the bimolecular macrocycle. (*c*) The two-dimensional network linked by C1—H1B···O1^{iii} and C23—H23···F1^{ii} hydrogen bonds, indicated by dashed lines. Disordered atoms F1′, F2′ and F3′ have been omitted for clarity. [Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+1, -z$.]

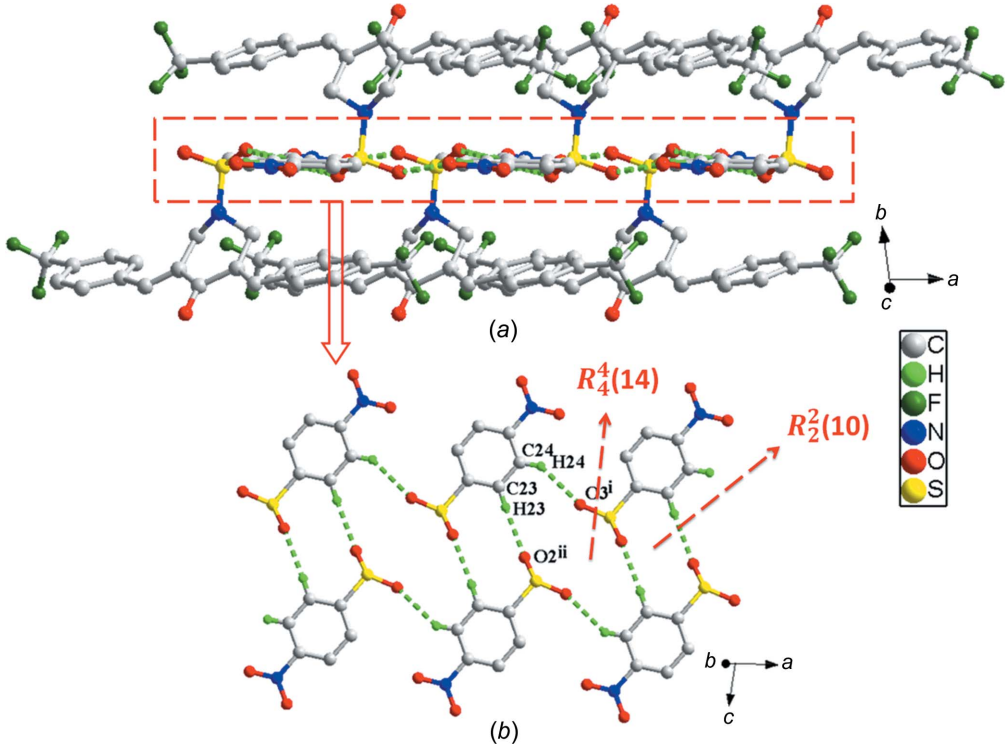


Figure 7
(*a*) The one-dimensional chain formed through intermolecular hydrogen bonds in (**6**). (*b*) The detailed hydrogen bonding, indicated by dashed lines. The 3,5-bis[4-(trifluoromethyl)benzylidene]piperidin-4-one groups have been omitted for clarity. [Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $x+1, y, z$.]

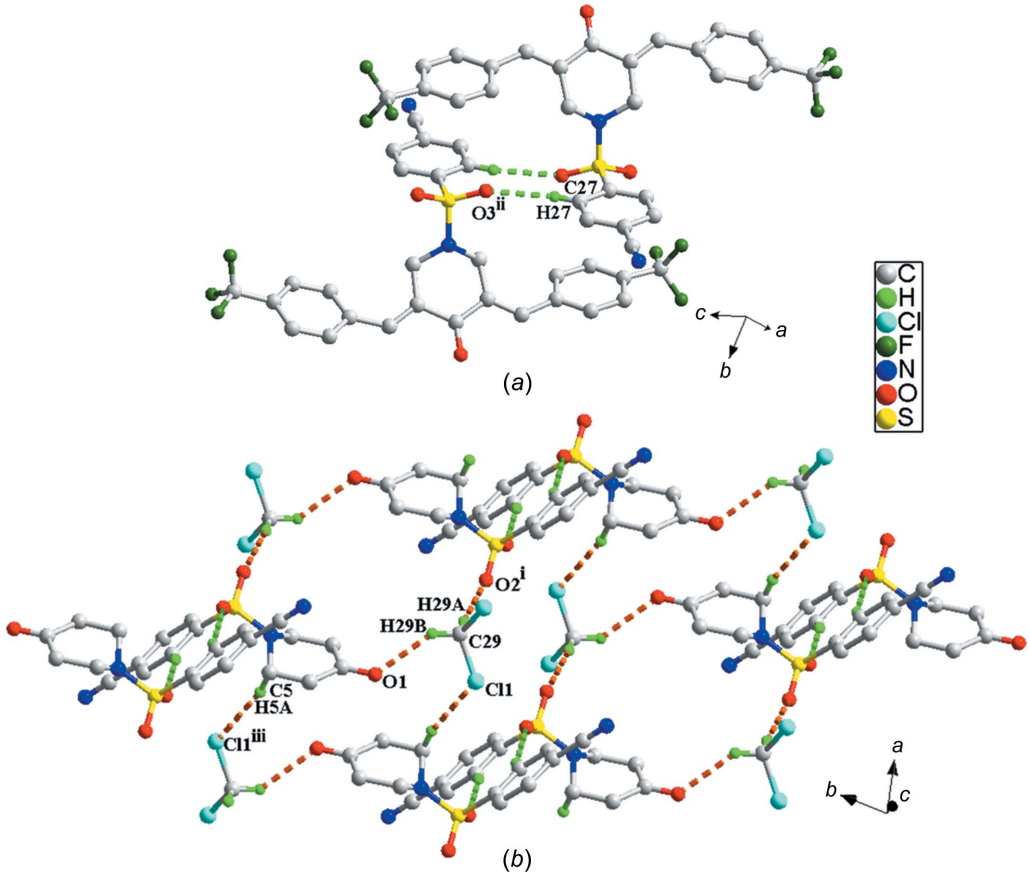


Figure 8
(*a*) The bimolecular macrocycle formed through intermolecular C27—H27···O3^{ii} hydrogen bonds in (**7**). (*b*) The two-dimensional network linked by hydrogen bonds between molecules of dichloromethane and BAP in (**7**). The 4-(trifluoromethyl)benzylidene groups have been omitted for clarity. Dashed lines indicate hydrogen bonds. [Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+2, -z+1$; (iii) $-x, -y+1, -z+1$.]

Table 3
Hydrogen-bond geometry (Å, °) for (**5**).

D—H···A	D—H	H···A	D···A	D—H···A
C26—H26···O1^{i}	0.95	2.58	3.458 (3)	154
C23—H23···F2$'^{ii}$	0.95	2.58	3.364 (12)	140
C23—H23···F1^{ii}	0.95	2.61	3.242 (7)	124
C1—H1B···O1^{iii}	0.99	2.46	3.293 (3)	142

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+1, -z$.

Table 4
Hydrogen-bond geometry (Å, °) for (**6**).

D—H···A	D—H	H···A	D···A	D—H···A
C24—H24···O3^{i}	0.95	2.56	3.380 (4)	145
C23—H23···O2^{ii}	0.95	2.36	3.304 (3)	176

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+2$.

top view in Fig. 6(*b*), an open elliptic capsule exists, where the O1···O1^{i} separation is 5.390 (3) Å and the S1···S1^{i} separation is 11.134 (1) Å. In the solid state, as shown in Fig. 6(*c*), the bimolecular macrocycles are linked into a two-dimensional sheet by C1—H1B···O1^{iii} and C23—H23···F1^{ii} hydrogen bonds (see Table 3 for details and symmetry codes).

In (**6**), a one-dimensional chain is generated by intermolecular hydrogen bonds which extends in the crystallographic *a*-axis direction (Fig. 7*a*). 3,5-Bis[4-(trifluoromethyl)benzylidene]piperidin-4-one units are embedded in both sides of the one-dimensional chain *via* N—S bonds. For clarity, these units are omitted in Fig. 7(*b*). The chain looks like a planar ribbon. An $R_2^2(10)$ motif and an $R_4^4(14)$ motif are formed from C23—H23···O2^{ii} and C24—H24···O3^{i} hydrogen bonds (Etter, 1990). Relevant hydrogen-bonding geometries and symmetry codes are given in Table 4.

As shown in Fig. 8(*a*), and different from (**5**), two BAP molecules of (**7**) are joined together *via* intermolecular C27—H27···O3^{ii} hydrogen bonds to generate a bimolecular combination without a macrocycle. An $R_2^2(10)$ motif can be found in the bimolecular combination. The dichloromethane solvent molecule plays a key role in the crystal packing. Two hydrogen-bonding donors (C—H) are joined to two groups of bimolecular combination through C29—H29B···O1 and C29—H29A···O2^{i} hydrogen bonds. Meanwhile, the hydrogen-bonding acceptor (Cl atom) links to a third bimolecular combination *via* C5—H5A···Cl1^{iii} hydrogen bonds. The bimolecular combinations are thus extended into a two-dimensional network (Fig. 8*b*). Relevant hydrogen-bonding geometries and symmetry codes are given in Table 5.

Table 5
Hydrogen-bond geometry (Å, °) for (**7**).

D—H···A	D—H	H···A	D···A	D—H···A
C29—H29B···O1	0.99	2.57	3.342 (4)	135
C29—H29A···O2^{i}	0.99	2.53	3.500 (4)	166
C27—H27···O3^{ii}	0.95	2.48	3.362 (3)	155
C5—H5A···Cl1^{iii}	0.99	2.81	3.776 (3)	166

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+2, -z+1$; (iii) $-x, -y+1, -z+1$.

The distinct structural features of the four title compounds are the different substituents (–CH_3, –F, –NO_2 and –CN) on the *N*-benzenesulfonyl group of (**4**)–(**7**). These substituents do not play a major role in the hydrogen-bonding patterns and crystal packing. However, their electron-donating or electron-withdrawing effect may influence biological activity.

3.4. Anti-inflammatory activity

LPS is a main component of Gram-negative bacterial endotoxin and can directly activate macrophages to secrete pro-inflammatory cytokines, such as TNF-α and IL-6 (Singh *et al.*, 2017). In our study, (**4**)–(**7**) had no significant toxicity at 10 µ*M* on RAW264.7 cells (Fig. 9). The effect of the four compounds on IL-6 and TNF-α secretion in RAW264.7 cells stimulated by LPS was detected by ELISA. PDTC was used as a reference standard. After treatment with (**4**)–(**7**), the secretion of IL-6 and TNF-α was significantly decreased with a 35–70% inhibition rate at a concentration of 10 µ*M* compared with only LPS-stimulated cells; the inhibitory effects are

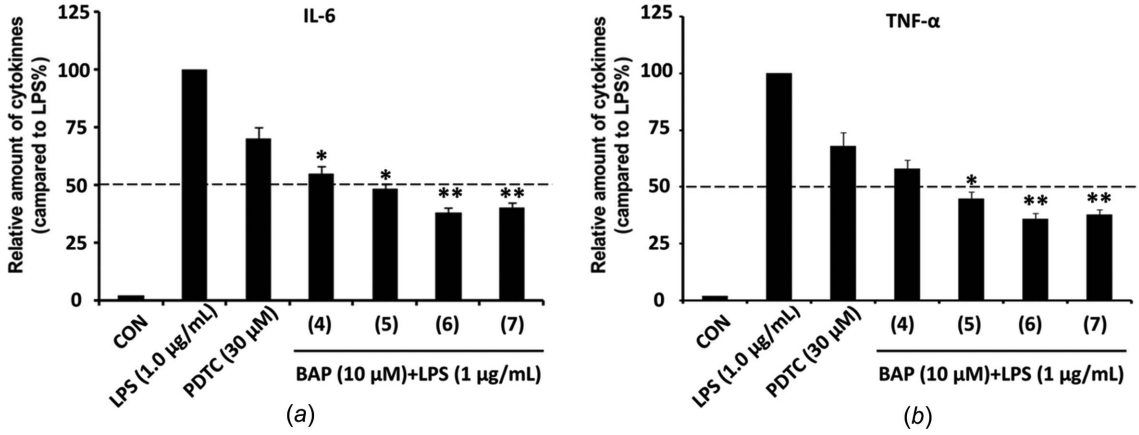


Figure 9
(*a*) IL-6 expression levels in the culture media measured by ELISA. (*b*) TNF-α expression levels in the culture media tested by ELISA. PDTC was used as a positive control. The results were presented as the percent of LPS control. Each bar represents the mean ±SD of three independent experiments. Statistical significance relative to the LPS group is indicated: (*) $p < 0.05$; (**) $p < 0.01$.

superior to PDTC. In particular, (**6**) and (**7**), with strong electron-withdrawing substitutes (–NO_2 and –CN), lead to a significant inhibitory effect against both TNF-α and IL-6 secretion compared with (**4**), having an electron-donating group (–CH_3), and (**5**), having a weak electron-withdrawing substituent (–F).

Funding information

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supporting information

Acta Cryst. (2018). C**74**, 1171-1179 [https://doi.org/10.1107/S2053229618013232]

Synthesis, crystal structures and anti-inflammatory activity of four 3,5-bis(arylidene)-*N*-benzenesulfonyl-4-piperidone derivatives

Ning Li, Xianyong Bai, Lianshuang Zhang and Yun Hou

Computing details

For all structures, data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(3*E*,5*E*)-1-(4-Fluorobenzenesulfonyl)-3,5-bis[4-(trifluoromethyl)benzylidene]piperidin-4-one dichloromethane monosolvate (4)

Crystal data

$C_{28}H_{21}F_6NO_3S\cdot CH_2Cl_2$
$M_r = 650.44$
Monoclinic, $P2_1/n$
$a = 8.6718$ (5) Å
$b = 23.5083$ (12) Å
$c = 14.3939$ (9) Å
$\beta = 102.962$ (2)°
$V = 2859.6$ (3) Å^3
$Z = 4$
$F(000) = 1328$
$D_x = 1.511$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9943 reflections
$\theta = 2.5$–$26.4°$
$\mu = 0.37$ mm^{-1}
$T = 150$ K
Block, yellow
0.14 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: sealed tube
phi and ω scans
38920 measured reflections
5852 independent reflections
4236 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.3°$
$h = -10\rightarrow10$
$k = -29\rightarrow29$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.126$
$S = 1.04$
5852 reflections
408 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0541P)^2 + 2.6939P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.69$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.6042 (2)	0.09574 (8)	0.02605 (13)	0.0343 (4)	
C1	0.2119 (3)	0.02787 (10)	0.00437 (17)	0.0224 (5)	
H1A	0.2169	−0.0110	−0.0211	0.027*	
H1B	0.1468	0.0265	0.0527	0.027*	
C2	0.3771 (3)	0.04750 (9)	0.05106 (16)	0.0210 (5)	
C3	0.4690 (3)	0.07967 (10)	−0.00795 (17)	0.0230 (5)	
C4	0.3932 (3)	0.09152 (10)	−0.11067 (17)	0.0218 (5)	
C5	0.2261 (3)	0.07064 (10)	−0.14871 (17)	0.0227 (5)	
H5A	0.1707	0.0971	−0.1986	0.027*	
H5B	0.2296	0.0329	−0.1785	0.027*	
C6	0.4445 (3)	0.04187 (10)	0.14446 (17)	0.0226 (5)	
H6	0.5488	0.0566	0.1639	0.027*	
C7	0.3796 (3)	0.01625 (10)	0.22028 (17)	0.0229 (5)	
C8	0.4273 (3)	0.03902 (11)	0.31209 (18)	0.0278 (5)	
H8	0.5014	0.0694	0.3232	0.033*	
C9	0.3683 (3)	0.01808 (11)	0.38682 (18)	0.0287 (5)	
H9	0.3993	0.0348	0.4483	0.034*	
C10	0.2636 (3)	−0.02744 (10)	0.37199 (18)	0.0260 (5)	
C11	0.2186 (3)	−0.05167 (10)	0.28188 (17)	0.0253 (5)	
H11	0.1489	−0.0834	0.2720	0.030*	
C12	0.2746 (3)	−0.02990 (10)	0.20634 (17)	0.0234 (5)	
H12	0.2418	−0.0463	0.1448	0.028*	
C13	0.2037 (3)	−0.05211 (11)	0.45243 (19)	0.0332 (6)	
C14	0.4793 (3)	0.12141 (10)	−0.16082 (17)	0.0238 (5)	
H14	0.5802	0.1337	−0.1262	0.029*	
C15	0.4379 (3)	0.13748 (10)	−0.26189 (17)	0.0254 (5)	
C16	0.5050 (3)	0.18706 (12)	−0.28977 (19)	0.0351 (6)	
H16	0.5766	0.2086	−0.2434	0.042*	
C17	0.4692 (4)	0.20519 (12)	−0.3833 (2)	0.0391 (7)	
H17	0.5156	0.2389	−0.4010	0.047*	
C18	0.3644 (3)	0.17365 (11)	−0.45169 (18)	0.0308 (6)	
C19	0.3013 (3)	0.12340 (10)	−0.42675 (18)	0.0278 (5)	
H19	0.2331	0.1013	−0.4740	0.033*	
C20	0.3378 (3)	0.10541 (10)	−0.33248 (17)	0.0253 (5)	
H20	0.2943	0.0708	−0.3157	0.030*	
C21	0.3248 (4)	0.19397 (14)	−0.5530 (2)	0.0449 (7)	
C22	0.1810 (3)	0.17179 (9)	0.01432 (16)	0.0209 (5)	
C23	0.2330 (3)	0.16704 (10)	0.11316 (17)	0.0228 (5)	
H23	0.1926	0.1380	0.1470	0.027*	

C24	0.3443 (3)	0.20520 (10)	0.16107 (17)	0.0257 (5)	
H24	0.3798	0.2022	0.2283	0.031*	
C25	0.4053 (3)	0.24791 (10)	0.11270 (19)	0.0291 (6)	
C26	0.3515 (3)	0.25165 (10)	0.01411 (19)	0.0312 (6)	
H26	0.3920	0.2807	−0.0197	0.037*	
C27	0.2402 (3)	0.21396 (10)	−0.03562 (17)	0.0263 (5)	
H27	0.2049	0.2169	−0.1029	0.032*	
C28	0.5273 (4)	0.28894 (13)	0.1653 (2)	0.0435 (7)	
H28A	0.5058	0.2972	0.2280	0.065*	
H28B	0.5227	0.3243	0.1287	0.065*	
H28C	0.6328	0.2721	0.1736	0.065*	
C29	0.8703 (3)	0.13523 (12)	0.2147 (2)	0.0350 (6)	
H29A	0.8038	0.1310	0.1495	0.042*	
H29B	0.9529	0.1639	0.2124	0.042*	
Cl1	0.96073 (11)	0.06994 (3)	0.25227 (7)	0.0580 (2)	
Cl2	0.75207 (10)	0.15934 (4)	0.29150 (6)	0.0525 (2)	
F1	0.2185 (2)	−0.01731 (8)	0.52703 (12)	0.0533 (5)	
F2	0.0510 (2)	−0.06637 (10)	0.42708 (13)	0.0611 (6)	
F3	0.2815 (3)	−0.09948 (8)	0.48702 (14)	0.0662 (6)	
F4	0.193 (3)	0.1743 (11)	−0.6004 (16)	0.106 (7)	0.51 (3)
F5	0.4359 (17)	0.1789 (8)	−0.5972 (8)	0.107 (4)	0.51 (3)
F6	0.3157 (16)	0.2480 (4)	−0.5636 (7)	0.064 (3)	0.51 (3)
F4′	0.255 (4)	0.2452 (7)	−0.5575 (10)	0.127 (6)	0.49 (3)
F5′	0.226 (3)	0.1614 (10)	−0.6123 (15)	0.087 (6)	0.49 (3)
F6′	0.4461 (11)	0.1994 (9)	−0.5910 (7)	0.114 (6)	0.49 (3)
N1	0.1363 (2)	0.06618 (8)	−0.07383 (13)	0.0197 (4)	
O2	−0.0492 (2)	0.10275 (8)	0.01773 (13)	0.0325 (4)	
O3	−0.0339 (2)	0.14734 (7)	−0.13659 (13)	0.0317 (4)	
S1	0.04238 (7)	0.12239 (2)	−0.04711 (4)	0.02246 (15)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0214 (9)	0.0512 (12)	0.0287 (10)	−0.0091 (8)	0.0025 (8)	0.0042 (8)
C1	0.0196 (11)	0.0220 (11)	0.0243 (12)	−0.0020 (9)	0.0021 (9)	0.0036 (9)
C2	0.0206 (11)	0.0186 (11)	0.0233 (12)	0.0011 (9)	0.0038 (9)	−0.0001 (9)
C3	0.0195 (12)	0.0255 (12)	0.0246 (13)	0.0025 (9)	0.0061 (10)	−0.0029 (9)
C4	0.0199 (11)	0.0233 (11)	0.0223 (12)	0.0017 (9)	0.0049 (9)	−0.0017 (9)
C5	0.0218 (12)	0.0267 (12)	0.0200 (12)	−0.0014 (9)	0.0053 (9)	−0.0037 (9)
C6	0.0200 (11)	0.0214 (11)	0.0264 (13)	−0.0006 (9)	0.0048 (10)	−0.0001 (9)
C7	0.0208 (11)	0.0234 (12)	0.0236 (13)	0.0020 (9)	0.0032 (10)	0.0031 (9)
C8	0.0271 (13)	0.0273 (12)	0.0272 (13)	−0.0062 (10)	0.0024 (10)	−0.0008 (10)
C9	0.0323 (14)	0.0314 (13)	0.0223 (13)	−0.0063 (11)	0.0056 (11)	−0.0041 (10)
C10	0.0242 (12)	0.0274 (12)	0.0259 (13)	0.0007 (10)	0.0049 (10)	0.0022 (10)
C11	0.0237 (12)	0.0236 (12)	0.0270 (13)	−0.0010 (10)	0.0027 (10)	0.0007 (10)
C12	0.0226 (12)	0.0246 (12)	0.0214 (12)	0.0024 (9)	0.0017 (9)	−0.0003 (9)
C13	0.0334 (14)	0.0363 (15)	0.0304 (14)	−0.0065 (12)	0.0083 (12)	−0.0015 (11)
C14	0.0203 (11)	0.0254 (12)	0.0247 (13)	0.0003 (9)	0.0030 (10)	−0.0016 (10)

C15	0.0238 (12)	0.0291 (13)	0.0247 (13)	−0.0010 (10)	0.0082 (10)	0.0014 (10)
C16	0.0389 (15)	0.0356 (14)	0.0299 (14)	−0.0155 (12)	0.0056 (12)	−0.0001 (11)
C17	0.0461 (17)	0.0375 (15)	0.0332 (15)	−0.0174 (13)	0.0081 (13)	0.0055 (12)
C18	0.0353 (14)	0.0339 (14)	0.0239 (13)	−0.0066 (11)	0.0085 (11)	0.0036 (11)
C19	0.0326 (13)	0.0267 (12)	0.0253 (13)	−0.0053 (11)	0.0092 (11)	−0.0030 (10)
C20	0.0287 (13)	0.0211 (11)	0.0279 (13)	−0.0028 (10)	0.0101 (10)	−0.0020 (10)
C21	0.054 (2)	0.0446 (18)	0.0345 (16)	−0.0121 (15)	0.0066 (15)	0.0090 (14)
C22	0.0201 (11)	0.0204 (11)	0.0213 (12)	0.0020 (9)	0.0031 (9)	−0.0038 (9)
C23	0.0252 (12)	0.0220 (11)	0.0220 (12)	0.0018 (9)	0.0068 (10)	0.0008 (9)
C24	0.0276 (13)	0.0301 (13)	0.0182 (12)	0.0032 (10)	0.0027 (10)	−0.0028 (10)
C25	0.0314 (13)	0.0257 (12)	0.0291 (14)	−0.0028 (10)	0.0042 (11)	−0.0059 (10)
C26	0.0393 (15)	0.0245 (12)	0.0304 (14)	−0.0080 (11)	0.0089 (12)	0.0011 (10)
C27	0.0336 (14)	0.0240 (12)	0.0196 (12)	−0.0018 (10)	0.0025 (10)	0.0004 (9)
C28	0.0471 (18)	0.0382 (16)	0.0407 (17)	−0.0171 (13)	0.0006 (14)	−0.0076 (13)
C29	0.0333 (14)	0.0382 (15)	0.0343 (15)	0.0004 (12)	0.0092 (12)	−0.0020 (12)
Cl1	0.0552 (5)	0.0386 (4)	0.0731 (6)	0.0080 (4)	−0.0008 (4)	−0.0017 (4)
Cl2	0.0503 (5)	0.0603 (5)	0.0525 (5)	−0.0012 (4)	0.0233 (4)	−0.0152 (4)
F1	0.0740 (13)	0.0572 (11)	0.0358 (10)	−0.0242 (10)	0.0276 (9)	−0.0126 (8)
F2	0.0443 (11)	0.0990 (16)	0.0428 (11)	−0.0341 (10)	0.0158 (9)	−0.0047 (10)
F3	0.0966 (16)	0.0520 (11)	0.0606 (13)	0.0175 (11)	0.0397 (12)	0.0311 (10)
F4	0.091 (7)	0.149 (16)	0.050 (9)	−0.078 (10)	−0.043 (7)	0.053 (8)
F5	0.137 (10)	0.161 (8)	0.040 (4)	0.075 (8)	0.054 (6)	0.035 (4)
F6	0.111 (6)	0.034 (3)	0.037 (4)	−0.020 (4)	−0.006 (4)	0.016 (2)
F4′	0.207 (15)	0.112 (10)	0.070 (5)	0.076 (9)	0.045 (8)	0.053 (6)
F5′	0.138 (17)	0.077 (5)	0.030 (3)	−0.065 (7)	−0.018 (6)	0.008 (3)
F6′	0.062 (5)	0.245 (14)	0.030 (4)	−0.085 (9)	−0.002 (4)	0.032 (6)
N1	0.0176 (9)	0.0213 (10)	0.0196 (10)	−0.0010 (7)	0.0028 (8)	0.0006 (8)
O2	0.0223 (9)	0.0379 (10)	0.0407 (11)	−0.0069 (8)	0.0143 (8)	−0.0092 (8)
O3	0.0296 (10)	0.0300 (9)	0.0290 (10)	0.0046 (7)	−0.0074 (8)	−0.0020 (8)
S1	0.0173 (3)	0.0243 (3)	0.0244 (3)	0.0002 (2)	0.0017 (2)	−0.0038 (2)

Geometric parameters (Å, º)

O1—C3	1.225 (3)	C17—C18	1.394 (4)
C1—N1	1.475 (3)	C17—H17	0.9500
C1—C2	1.511 (3)	C18—C19	1.383 (3)
C1—H1A	0.9900	C18—C21	1.499 (4)
C1—H1B	0.9900	C19—C20	1.388 (3)
C2—C6	1.347 (3)	C19—H19	0.9500
C2—C3	1.494 (3)	C20—H20	0.9500
C3—C4	1.503 (3)	C21—F6	1.279 (10)
C4—C14	1.347 (3)	C21—F4	1.279 (19)
C4—C5	1.512 (3)	C21—F6′	1.296 (10)
C5—N1	1.468 (3)	C21—F5′	1.311 (18)
C5—H5A	0.9900	C21—F5	1.317 (9)
C5—H5B	0.9900	C21—F4′	1.342 (16)
C6—C7	1.465 (3)	C22—C27	1.389 (3)
C6—H6	0.9500	C22—C23	1.397 (3)

C7—C8	1.399 (3)	C22—S1	1.760 (2)
C7—C12	1.401 (3)	C23—C24	1.383 (3)
C8—C9	1.381 (3)	C23—H23	0.9500
C8—H8	0.9500	C24—C25	1.392 (4)
C9—C10	1.388 (3)	C24—H24	0.9500
C9—H9	0.9500	C25—C26	1.394 (4)
C10—C11	1.390 (3)	C25—C28	1.505 (4)
C10—C13	1.489 (4)	C26—C27	1.385 (3)
C11—C12	1.385 (3)	C26—H26	0.9500
C11—H11	0.9500	C27—H27	0.9500
C12—H12	0.9500	C28—H28A	0.9800
C13—F1	1.333 (3)	C28—H28B	0.9800
C13—F2	1.335 (3)	C28—H28C	0.9800
C13—F3	1.339 (3)	C29—Cl1	1.753 (3)
C14—C15	1.467 (3)	C29—Cl2	1.761 (3)
C14—H14	0.9500	C29—H29A	0.9900
C15—C20	1.399 (3)	C29—H29B	0.9900
C15—C16	1.401 (4)	N1—S1	1.6428 (19)
C16—C17	1.380 (4)	O2—S1	1.4309 (18)
C16—H16	0.9500	O3—S1	1.4346 (18)

N1—C1—C2	111.56 (18)	C19—C18—C17	120.3 (2)
N1—C1—H1A	109.3	C19—C18—C21	120.5 (2)
C2—C1—H1A	109.3	C17—C18—C21	119.2 (2)
N1—C1—H1B	109.3	C18—C19—C20	119.8 (2)
C2—C1—H1B	109.3	C18—C19—H19	120.1
H1A—C1—H1B	108.0	C20—C19—H19	120.1
C6—C2—C3	116.7 (2)	C19—C20—C15	120.9 (2)
C6—C2—C1	124.7 (2)	C19—C20—H20	119.5
C3—C2—C1	118.4 (2)	C15—C20—H20	119.5
O1—C3—C2	120.9 (2)	F6—C21—F4	105.8 (12)
O1—C3—C4	120.3 (2)	F6′—C21—F5′	104.9 (14)
C2—C3—C4	118.8 (2)	F6—C21—F5	104.0 (8)
C14—C4—C3	116.8 (2)	F4—C21—F5	107.9 (13)
C14—C4—C5	125.2 (2)	F6′—C21—F4′	106.8 (9)
C3—C4—C5	117.9 (2)	F5′—C21—F4′	105.7 (14)
N1—C5—C4	112.36 (19)	F6—C21—C18	115.3 (5)
N1—C5—H5A	109.1	F4—C21—C18	112.7 (11)
C4—C5—H5A	109.1	F6′—C21—C18	114.3 (6)
N1—C5—H5B	109.1	F5′—C21—C18	114.9 (11)
C4—C5—H5B	109.1	F5—C21—C18	110.6 (5)
H5A—C5—H5B	107.9	F4′—C21—C18	109.6 (7)
C2—C6—C7	129.1 (2)	C27—C22—C23	120.8 (2)
C2—C6—H6	115.4	C27—C22—S1	120.07 (18)
C7—C6—H6	115.4	C23—C22—S1	119.13 (18)
C8—C7—C12	118.4 (2)	C24—C23—C22	119.1 (2)
C8—C7—C6	117.7 (2)	C24—C23—H23	120.5
C12—C7—C6	123.9 (2)	C22—C23—H23	120.5

C9—C8—C7	121.1 (2)	C23—C24—C25	121.3 (2)
C9—C8—H8	119.4	C23—C24—H24	119.4
C7—C8—H8	119.4	C25—C24—H24	119.4
C8—C9—C10	119.9 (2)	C24—C25—C26	118.5 (2)
C8—C9—H9	120.0	C24—C25—C28	120.9 (2)
C10—C9—H9	120.0	C26—C25—C28	120.6 (2)
C9—C10—C11	119.8 (2)	C27—C26—C25	121.4 (2)
C9—C10—C13	120.7 (2)	C27—C26—H26	119.3
C11—C10—C13	119.5 (2)	C25—C26—H26	119.3
C12—C11—C10	120.4 (2)	C26—C27—C22	119.0 (2)
C12—C11—H11	119.8	C26—C27—H27	120.5
C10—C11—H11	119.8	C22—C27—H27	120.5
C11—C12—C7	120.3 (2)	C25—C28—H28A	109.5
C11—C12—H12	119.8	C25—C28—H28B	109.5
C7—C12—H12	119.8	H28A—C28—H28B	109.5
F1—C13—F2	106.5 (2)	C25—C28—H28C	109.5
F1—C13—F3	105.2 (2)	H28A—C28—H28C	109.5
F2—C13—F3	106.2 (2)	H28B—C28—H28C	109.5
F1—C13—C10	113.6 (2)	Cl1—C29—Cl2	111.82 (16)
F2—C13—C10	112.5 (2)	Cl1—C29—H29A	109.3
F3—C13—C10	112.3 (2)	Cl2—C29—H29A	109.3
C4—C14—C15	128.6 (2)	Cl1—C29—H29B	109.3
C4—C14—H14	115.7	Cl2—C29—H29B	109.3
C15—C14—H14	115.7	H29A—C29—H29B	107.9
C20—C15—C16	118.0 (2)	C5—N1—C1	112.70 (18)
C20—C15—C14	123.7 (2)	C5—N1—S1	119.13 (15)
C16—C15—C14	118.2 (2)	C1—N1—S1	118.11 (15)
C17—C16—C15	121.3 (2)	O2—S1—O3	120.40 (11)
C17—C16—H16	119.4	O2—S1—N1	105.67 (10)
C15—C16—H16	119.4	O3—S1—N1	105.75 (10)
C16—C17—C18	119.6 (2)	O2—S1—C22	107.56 (11)
C16—C17—H17	120.2	O3—S1—C22	107.85 (11)
C18—C17—H17	120.2	N1—S1—C22	109.25 (10)

N1—C1—C2—C6	145.7 (2)	C21—C18—C19—C20	−179.4 (3)
N1—C1—C2—C3	−29.1 (3)	C18—C19—C20—C15	0.1 (4)
C6—C2—C3—O1	5.9 (3)	C16—C15—C20—C19	−2.5 (4)
C1—C2—C3—O1	−178.8 (2)	C14—C15—C20—C19	179.0 (2)
C6—C2—C3—C4	−174.8 (2)	C19—C18—C21—F6	145.8 (7)
C1—C2—C3—C4	0.5 (3)	C17—C18—C21—F6	−35.9 (8)
O1—C3—C4—C14	−1.7 (3)	C19—C18—C21—F4	24.3 (17)
C2—C3—C4—C14	179.0 (2)	C17—C18—C21—F4	−157.5 (17)
O1—C3—C4—C5	180.0 (2)	C19—C18—C21—F6′	−119.9 (11)
C2—C3—C4—C5	0.6 (3)	C17—C18—C21—F6′	58.3 (11)
C14—C4—C5—N1	−150.9 (2)	C19—C18—C21—F5′	1.5 (14)
C3—C4—C5—N1	27.2 (3)	C17—C18—C21—F5′	179.7 (14)
C3—C2—C6—C7	175.6 (2)	C19—C18—C21—F5	−96.6 (11)
C1—C2—C6—C7	0.7 (4)	C17—C18—C21—F5	81.7 (11)

C2—C6—C7—C8	−147.9 (3)	C19—C18—C21—F4′	120.2 (15)
C2—C6—C7—C12	32.9 (4)	C17—C18—C21—F4′	−61.6 (15)
C12—C7—C8—C9	−2.4 (4)	C27—C22—C23—C24	0.3 (3)
C6—C7—C8—C9	178.4 (2)	S1—C22—C23—C24	178.72 (18)
C7—C8—C9—C10	2.0 (4)	C22—C23—C24—C25	−0.2 (4)
C8—C9—C10—C11	−0.1 (4)	C23—C24—C25—C26	0.1 (4)
C8—C9—C10—C13	177.5 (2)	C23—C24—C25—C28	−179.5 (2)
C9—C10—C11—C12	−1.5 (4)	C24—C25—C26—C27	−0.1 (4)
C13—C10—C11—C12	−179.1 (2)	C28—C25—C26—C27	179.5 (3)
C10—C11—C12—C7	1.1 (4)	C25—C26—C27—C22	0.3 (4)
C8—C7—C12—C11	0.8 (3)	C23—C22—C27—C26	−0.3 (4)
C6—C7—C12—C11	−180.0 (2)	S1—C22—C27—C26	−178.74 (19)
C9—C10—C13—F1	19.5 (4)	C4—C5—N1—C1	−58.1 (2)
C11—C10—C13—F1	−162.9 (2)	C4—C5—N1—S1	86.8 (2)
C9—C10—C13—F2	140.7 (3)	C2—C1—N1—C5	58.9 (2)
C11—C10—C13—F2	−41.7 (3)	C2—C1—N1—S1	−86.3 (2)
C9—C10—C13—F3	−99.6 (3)	C5—N1—S1—O2	172.46 (16)
C11—C10—C13—F3	78.0 (3)	C1—N1—S1—O2	−44.55 (18)
C3—C4—C14—C15	177.9 (2)	C5—N1—S1—O3	43.76 (19)
C5—C4—C14—C15	−3.9 (4)	C1—N1—S1—O3	−173.25 (16)
C4—C14—C15—C20	−29.5 (4)	C5—N1—S1—C22	−72.08 (18)
C4—C14—C15—C16	152.1 (3)	C1—N1—S1—C22	70.91 (18)
C20—C15—C16—C17	2.5 (4)	C27—C22—S1—O2	−153.82 (19)
C14—C15—C16—C17	−178.9 (3)	C23—C22—S1—O2	27.7 (2)
C15—C16—C17—C18	−0.1 (5)	C27—C22—S1—O3	−22.6 (2)
C16—C17—C18—C19	−2.4 (4)	C23—C22—S1—O3	159.00 (18)
C16—C17—C18—C21	179.4 (3)	C27—C22—S1—N1	91.9 (2)
C17—C18—C19—C20	2.4 (4)	C23—C22—S1—N1	−86.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O1^{i}	0.99	2.53	3.390 (3)	145
C1—H1*A*···O2^{ii}	0.99	2.61	3.365 (3)	133
C5—H5*B*···Cl1^{i}	0.99	2.98	3.832 (2)	144
C29—H29*A*···O1	0.99	2.34	3.279 (3)	159
C29—H29*A*···O2^{iii}	0.99	2.60	3.162 (3)	116

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) −*x*, −*y*, −*z*; (iii) *x*+1, *y*, *z*.

(3*E*,5*E*)-1-(4-Fluorobenzenesulfonyl)-3,5-bis[4-(trifluoromethyl)benzylidene]piperidin-4-one (5)

Crystal data

$C_{27}H_{18}F_7NO_3S$
M_r = 569.48
Triclinic, $P\overline{1}$
a = 8.5664 (6) Å
b = 10.0932 (8) Å
c = 14.3871 (12) Å
α = 90.590 (4)°
β = 98.592 (4)°
γ = 97.309 (4)°
V = 1219.44 (17) Å^3
Z = 2
F(000) = 580
D_x = 1.551 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å

Cell parameters from 4723 reflections
θ = 2.4–26.3°
μ = 0.22 mm^{-1}

T = 150 K
Block, yellow
0.26 × 0.17 × 0.10 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: sealed tube
phi and ω scans
21256 measured reflections
4790 independent reflections

3187 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 26.0°, θ_{min} = 2.4°
h = −10→10
k = −12→12
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.129
S = 1.01
4790 reflections
380 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.8089P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.2745 (3)	0.4646 (3)	0.09345 (19)	0.0266 (6)	
H1A	0.2746	0.4750	0.1620	0.032*	
H1B	0.2856	0.5549	0.0671	0.032*	
C2	0.4151 (3)	0.3950 (3)	0.07680 (18)	0.0229 (6)	
C3	0.3998 (3)	0.3125 (3)	−0.01181 (18)	0.0235 (6)	
C4	0.2444 (3)	0.2949 (3)	−0.07648 (18)	0.0233 (6)	
C5	0.1130 (3)	0.3682 (3)	−0.05191 (18)	0.0278 (6)	
H5A	0.1188	0.4558	−0.0823	0.033*	
H5B	0.0089	0.3163	−0.0770	0.033*	
C6	0.5502 (3)	0.3970 (3)	0.13772 (18)	0.0252 (6)	
H6	0.6278	0.3489	0.1172	0.030*	
C7	0.5962 (3)	0.4619 (3)	0.23108 (18)	0.0231 (6)	
C8	0.7029 (3)	0.4042 (3)	0.2959 (2)	0.0277 (6)	
H8	0.7437	0.3262	0.2778	0.033*	
C9	0.7510 (3)	0.4579 (3)	0.3861 (2)	0.0294 (6)	
H9	0.8232	0.4168	0.4296	0.035*	
C10	0.6926 (3)	0.5725 (3)	0.41233 (19)	0.0260 (6)	
C11	0.5903 (3)	0.6343 (3)	0.34794 (19)	0.0271 (6)	
H11	0.5529	0.7141	0.3656	0.032*	
C12	0.5429 (3)	0.5799 (3)	0.25799 (19)	0.0249 (6)	

H12	0.4735	0.6231	0.2140	0.030*	
C13	0.7432 (4)	0.6305 (3)	0.5094 (2)	0.0331 (7)	
C14	0.2319 (3)	0.2154 (3)	−0.15292 (19)	0.0252 (6)	
H14	0.3198	0.1678	−0.1564	0.030*	
C15	0.1018 (3)	0.1914 (3)	−0.23187 (18)	0.0254 (6)	
C16	0.0801 (3)	0.0706 (3)	−0.2831 (2)	0.0328 (7)	
H16	0.1465	0.0043	−0.2639	0.039*	
C17	−0.0355 (3)	0.0453 (3)	−0.3610 (2)	0.0361 (7)	
H17	−0.0490	−0.0380	−0.3945	0.043*	
C18	−0.1322 (3)	0.1420 (3)	−0.3901 (2)	0.0316 (7)	
C19	−0.1114 (4)	0.2635 (3)	−0.3416 (2)	0.0349 (7)	
H19	−0.1765	0.3303	−0.3621	0.042*	
C20	0.0040 (3)	0.2877 (3)	−0.26338 (19)	0.0298 (6)	
H20	0.0173	0.3714	−0.2303	0.036*	
C21	−0.2521 (4)	0.1177 (4)	−0.4773 (2)	0.0420 (8)	
C22	0.1738 (3)	0.1683 (3)	0.15526 (18)	0.0225 (6)	
C23	0.2648 (3)	0.1949 (3)	0.24334 (19)	0.0277 (6)	
H23	0.2506	0.2695	0.2805	0.033*	
C24	0.3765 (3)	0.1122 (3)	0.2766 (2)	0.0318 (7)	
H24	0.4408	0.1290	0.3363	0.038*	
C25	0.3913 (3)	0.0050 (3)	0.2207 (2)	0.0324 (7)	
C26	0.3034 (3)	−0.0243 (3)	0.1338 (2)	0.0332 (7)	
H26	0.3173	−0.1000	0.0976	0.040*	
C27	0.1933 (3)	0.0600 (3)	0.1001 (2)	0.0283 (6)	
H27	0.1315	0.0436	0.0396	0.034*	
F1	0.6329 (10)	0.6923 (9)	0.5414 (5)	0.061 (3)	0.542 (19)
F2	0.763 (2)	0.5383 (5)	0.5743 (3)	0.077 (4)	0.542 (19)
F3	0.8690 (9)	0.7143 (13)	0.5196 (5)	0.076 (4)	0.542 (19)
F3′	0.8703 (14)	0.5860 (14)	0.5537 (7)	0.079 (5)	0.458 (19)
F2′	0.6354 (14)	0.613 (2)	0.5590 (7)	0.119 (8)	0.458 (19)
F1′	0.783 (3)	0.7594 (7)	0.5101 (6)	0.096 (6)	0.458 (19)
F4	−0.3734 (2)	0.1880 (3)	−0.47810 (15)	0.0800 (8)	
F5	−0.3093 (3)	−0.0095 (2)	−0.49351 (16)	0.0797 (8)	
F6	−0.1910 (2)	0.1550 (2)	−0.55509 (12)	0.0499 (5)	
F7	0.4980 (2)	−0.0776 (2)	0.25495 (13)	0.0534 (5)	
N1	0.1225 (2)	0.3894 (2)	0.04994 (15)	0.0243 (5)	
O1	0.5140 (2)	0.26338 (19)	−0.03256 (13)	0.0310 (5)	
O2	0.0046 (2)	0.3482 (2)	0.19369 (14)	0.0369 (5)	
O3	−0.0936 (2)	0.2044 (2)	0.05130 (15)	0.0384 (5)	
S1	0.03566 (8)	0.27738 (7)	0.11325 (5)	0.02732 (19)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0285 (14)	0.0253 (15)	0.0277 (15)	0.0074 (12)	0.0068 (12)	−0.0020 (12)
C2	0.0271 (14)	0.0199 (14)	0.0241 (14)	0.0063 (11)	0.0093 (11)	0.0023 (11)
C3	0.0266 (14)	0.0219 (14)	0.0242 (14)	0.0067 (11)	0.0075 (11)	0.0031 (11)
C4	0.0254 (14)	0.0235 (14)	0.0236 (14)	0.0083 (11)	0.0081 (11)	0.0038 (11)

C5	0.0301 (15)	0.0319 (16)	0.0235 (14)	0.0126 (12)	0.0038 (12)	−0.0006 (12)
C6	0.0267 (14)	0.0237 (15)	0.0281 (15)	0.0076 (11)	0.0101 (12)	0.0014 (11)
C7	0.0229 (13)	0.0238 (14)	0.0240 (14)	0.0034 (11)	0.0075 (11)	0.0031 (11)
C8	0.0280 (14)	0.0242 (15)	0.0329 (16)	0.0068 (12)	0.0081 (12)	0.0005 (12)
C9	0.0286 (15)	0.0306 (16)	0.0289 (16)	0.0066 (12)	0.0016 (12)	0.0035 (12)
C10	0.0256 (14)	0.0265 (15)	0.0256 (15)	0.0006 (12)	0.0056 (12)	0.0027 (12)
C11	0.0310 (15)	0.0236 (15)	0.0280 (15)	0.0062 (12)	0.0066 (12)	0.0002 (12)
C12	0.0271 (14)	0.0223 (14)	0.0251 (15)	0.0042 (11)	0.0028 (11)	0.0021 (11)
C13	0.0379 (17)	0.0293 (17)	0.0307 (16)	0.0036 (14)	0.0008 (14)	−0.0011 (13)
C14	0.0229 (13)	0.0248 (15)	0.0296 (15)	0.0049 (11)	0.0080 (11)	0.0011 (12)
C15	0.0251 (14)	0.0283 (15)	0.0238 (14)	0.0006 (12)	0.0091 (11)	0.0004 (12)
C16	0.0265 (14)	0.0358 (17)	0.0362 (17)	0.0043 (13)	0.0056 (13)	−0.0052 (14)
C17	0.0311 (16)	0.0358 (18)	0.0403 (18)	−0.0006 (13)	0.0065 (14)	−0.0104 (14)
C18	0.0250 (14)	0.0378 (18)	0.0307 (16)	−0.0044 (13)	0.0068 (12)	0.0026 (13)
C19	0.0368 (17)	0.0368 (18)	0.0317 (17)	0.0074 (14)	0.0049 (14)	0.0088 (14)
C20	0.0361 (16)	0.0278 (15)	0.0257 (15)	0.0034 (13)	0.0062 (13)	0.0016 (12)
C21	0.0342 (17)	0.049 (2)	0.0399 (19)	−0.0007 (15)	0.0025 (15)	−0.0023 (16)
C22	0.0219 (13)	0.0216 (14)	0.0252 (14)	0.0042 (11)	0.0063 (11)	0.0030 (11)
C23	0.0332 (15)	0.0277 (15)	0.0234 (15)	0.0067 (12)	0.0059 (12)	0.0005 (12)
C24	0.0353 (16)	0.0363 (17)	0.0243 (15)	0.0083 (13)	0.0024 (12)	0.0058 (13)
C25	0.0302 (15)	0.0339 (17)	0.0368 (17)	0.0148 (13)	0.0078 (13)	0.0082 (13)
C26	0.0397 (17)	0.0276 (16)	0.0351 (17)	0.0115 (13)	0.0091 (14)	−0.0018 (13)
C27	0.0298 (15)	0.0265 (15)	0.0287 (15)	0.0048 (12)	0.0042 (12)	−0.0023 (12)
F1	0.065 (5)	0.091 (6)	0.031 (3)	0.043 (5)	−0.002 (3)	−0.022 (3)
F2	0.162 (12)	0.037 (3)	0.026 (2)	0.024 (4)	−0.011 (4)	0.0051 (18)
F3	0.061 (4)	0.104 (10)	0.052 (4)	−0.047 (4)	0.019 (3)	−0.036 (5)
F3′	0.081 (6)	0.095 (9)	0.055 (5)	0.055 (6)	−0.044 (4)	−0.033 (5)
F2′	0.069 (8)	0.23 (2)	0.041 (5)	−0.071 (10)	0.033 (5)	−0.045 (9)
F1′	0.186 (16)	0.032 (3)	0.050 (4)	−0.012 (5)	−0.029 (7)	−0.008 (3)
F4	0.0400 (12)	0.141 (2)	0.0594 (14)	0.0360 (13)	−0.0095 (10)	−0.0291 (15)
F5	0.0899 (17)	0.0570 (15)	0.0673 (15)	−0.0307 (13)	−0.0355 (13)	0.0098 (12)
F6	0.0537 (12)	0.0604 (13)	0.0344 (11)	0.0093 (10)	0.0014 (9)	0.0023 (9)
F7	0.0586 (12)	0.0606 (13)	0.0484 (12)	0.0399 (10)	0.0041 (9)	0.0096 (10)
N1	0.0237 (11)	0.0269 (13)	0.0245 (12)	0.0085 (10)	0.0061 (9)	−0.0005 (10)
O1	0.0285 (10)	0.0352 (11)	0.0319 (11)	0.0138 (9)	0.0060 (8)	−0.0062 (9)
O2	0.0414 (12)	0.0437 (13)	0.0338 (12)	0.0190 (10)	0.0206 (9)	0.0030 (10)
O3	0.0212 (10)	0.0460 (13)	0.0455 (13)	0.0031 (9)	−0.0021 (9)	0.0019 (10)
S1	0.0225 (3)	0.0323 (4)	0.0300 (4)	0.0100 (3)	0.0079 (3)	0.0017 (3)

Geometric parameters (Å, º)

C1—N1	1.469 (3)	C14—C15	1.462 (4)
C1—C2	1.516 (3)	C14—H14	0.9500
C1—H1A	0.9900	C15—C16	1.396 (4)
C1—H1B	0.9900	C15—C20	1.398 (4)
C2—C6	1.341 (4)	C16—C17	1.378 (4)
C2—C3	1.495 (4)	C16—H16	0.9500
C3—O1	1.225 (3)	C17—C18	1.388 (4)

C3—C4	1.495 (4)	C17—H17	0.9500
C4—C14	1.338 (4)	C18—C19	1.383 (4)
C4—C5	1.505 (3)	C18—C21	1.494 (4)
C5—N1	1.467 (3)	C19—C20	1.380 (4)
C5—H5A	0.9900	C19—H19	0.9500
C5—H5B	0.9900	C20—H20	0.9500
C6—C7	1.465 (4)	C21—F5	1.320 (4)
C6—H6	0.9500	C21—F4	1.330 (4)
C7—C8	1.391 (4)	C21—F6	1.343 (4)
C7—C12	1.400 (4)	C22—C27	1.386 (4)
C8—C9	1.384 (4)	C22—C23	1.390 (4)
C8—H8	0.9500	C22—S1	1.763 (2)
C9—C10	1.387 (4)	C23—C24	1.384 (4)
C9—H9	0.9500	C23—H23	0.9500
C10—C11	1.387 (4)	C24—C25	1.372 (4)
C10—C13	1.489 (4)	C24—H24	0.9500
C11—C12	1.383 (4)	C25—F7	1.357 (3)
C11—H11	0.9500	C25—C26	1.368 (4)
C12—H12	0.9500	C26—C27	1.387 (4)
C13—F2′	1.246 (9)	C26—H26	0.9500
C13—F3	1.272 (7)	C27—H27	0.9500
C13—F1′	1.303 (8)	N1—S1	1.634 (2)
C13—F3′	1.310 (6)	O2—S1	1.430 (2)
C13—F1	1.331 (7)	O3—S1	1.431 (2)
C13—F2	1.333 (5)		

N1—C1—C2	111.8 (2)	C4—C14—C15	129.0 (2)
N1—C1—H1A	109.3	C4—C14—H14	115.5
C2—C1—H1A	109.3	C15—C14—H14	115.5
N1—C1—H1B	109.3	C16—C15—C20	117.6 (3)
C2—C1—H1B	109.3	C16—C15—C14	119.0 (2)
H1A—C1—H1B	107.9	C20—C15—C14	123.2 (3)
C6—C2—C3	117.2 (2)	C17—C16—C15	121.5 (3)
C6—C2—C1	124.8 (2)	C17—C16—H16	119.2
C3—C2—C1	117.9 (2)	C15—C16—H16	119.2
O1—C3—C4	120.4 (2)	C16—C17—C18	119.6 (3)
O1—C3—C2	120.6 (2)	C16—C17—H17	120.2
C4—C3—C2	119.0 (2)	C18—C17—H17	120.2
C14—C4—C3	117.6 (2)	C19—C18—C17	120.0 (3)
C14—C4—C5	124.3 (2)	C19—C18—C21	120.1 (3)
C3—C4—C5	118.1 (2)	C17—C18—C21	119.8 (3)
N1—C5—C4	112.3 (2)	C20—C19—C18	119.9 (3)
N1—C5—H5A	109.1	C20—C19—H19	120.0
C4—C5—H5A	109.1	C18—C19—H19	120.0
N1—C5—H5B	109.1	C19—C20—C15	121.2 (3)
C4—C5—H5B	109.1	C19—C20—H20	119.4
H5A—C5—H5B	107.9	C15—C20—H20	119.4
C2—C6—C7	129.8 (2)	F5—C21—F4	108.6 (3)

C2—C6—H6	115.1	F5—C21—F6	104.6 (3)
C7—C6—H6	115.1	F4—C21—F6	103.8 (3)
C8—C7—C12	118.2 (2)	F5—C21—C18	113.4 (3)
C8—C7—C6	117.7 (2)	F4—C21—C18	113.2 (3)
C12—C7—C6	124.1 (2)	F6—C21—C18	112.5 (3)
C9—C8—C7	121.5 (2)	C27—C22—C23	120.9 (2)
C9—C8—H8	119.3	C27—C22—S1	120.3 (2)
C7—C8—H8	119.3	C23—C22—S1	118.8 (2)
C8—C9—C10	119.3 (2)	C24—C23—C22	119.7 (3)
C8—C9—H9	120.3	C24—C23—H23	120.2
C10—C9—H9	120.3	C22—C23—H23	120.2
C11—C10—C9	120.2 (3)	C25—C24—C23	117.9 (3)
C11—C10—C13	120.2 (2)	C25—C24—H24	121.0
C9—C10—C13	119.6 (2)	C23—C24—H24	121.0
C12—C11—C10	120.1 (2)	F7—C25—C26	118.6 (3)
C12—C11—H11	120.0	F7—C25—C24	117.5 (3)
C10—C11—H11	120.0	C26—C25—C24	123.9 (3)
C11—C12—C7	120.6 (2)	C25—C26—C27	117.9 (3)
C11—C12—H12	119.7	C25—C26—H26	121.0
C7—C12—H12	119.7	C27—C26—H26	121.0
F2′—C13—F1′	105.2 (8)	C22—C27—C26	119.6 (3)
F2′—C13—F3′	108.7 (7)	C22—C27—H27	120.2
F1′—C13—F3′	103.8 (6)	C26—C27—H27	120.2
F3—C13—F1	105.9 (5)	C5—N1—C1	112.9 (2)
F3—C13—F2	107.9 (5)	C5—N1—S1	120.87 (19)
F1—C13—F2	100.2 (5)	C1—N1—S1	117.40 (18)
F2′—C13—C10	112.7 (5)	O2—S1—O3	119.80 (12)
F3—C13—C10	114.8 (4)	O2—S1—N1	106.01 (12)
F1′—C13—C10	112.3 (4)	O3—S1—N1	106.49 (12)
F3′—C13—C10	113.5 (4)	O2—S1—C22	106.57 (12)
F1—C13—C10	113.5 (4)	O3—S1—C22	109.23 (13)
F2—C13—C10	113.3 (3)	N1—S1—C22	108.28 (11)

N1—C1—C2—C6	−146.5 (3)	C20—C15—C16—C17	−1.3 (4)
N1—C1—C2—C3	30.3 (3)	C14—C15—C16—C17	−176.7 (3)
C6—C2—C3—O1	−9.1 (4)	C15—C16—C17—C18	0.6 (5)
C1—C2—C3—O1	173.9 (2)	C16—C17—C18—C19	0.5 (4)
C6—C2—C3—C4	173.1 (2)	C16—C17—C18—C21	176.9 (3)
C1—C2—C3—C4	−4.0 (4)	C17—C18—C19—C20	−0.9 (4)
O1—C3—C4—C14	5.3 (4)	C21—C18—C19—C20	−177.3 (3)
C2—C3—C4—C14	−176.9 (2)	C18—C19—C20—C15	0.2 (4)
O1—C3—C4—C5	−174.3 (2)	C16—C15—C20—C19	0.9 (4)
C2—C3—C4—C5	3.5 (4)	C14—C15—C20—C19	176.2 (3)
C14—C4—C5—N1	150.8 (3)	C19—C18—C21—F5	−152.5 (3)
C3—C4—C5—N1	−29.6 (3)	C17—C18—C21—F5	31.1 (4)
C3—C2—C6—C7	−176.6 (3)	C19—C18—C21—F4	−28.2 (4)
C1—C2—C6—C7	0.2 (5)	C17—C18—C21—F4	155.4 (3)
C2—C6—C7—C8	151.6 (3)	C19—C18—C21—F6	89.0 (3)

C2—C6—C7—C12	−30.2 (5)	C17—C18—C21—F6	−87.4 (4)
C12—C7—C8—C9	2.6 (4)	C27—C22—C23—C24	0.0 (4)
C6—C7—C8—C9	−179.1 (3)	S1—C22—C23—C24	−178.5 (2)
C7—C8—C9—C10	−0.5 (4)	C22—C23—C24—C25	−0.7 (4)
C8—C9—C10—C11	−1.7 (4)	C23—C24—C25—F7	−178.3 (3)
C8—C9—C10—C13	179.7 (3)	C23—C24—C25—C26	0.5 (5)
C9—C10—C11—C12	1.7 (4)	F7—C25—C26—C27	179.2 (3)
C13—C10—C11—C12	−179.7 (3)	C24—C25—C26—C27	0.4 (5)
C10—C11—C12—C7	0.4 (4)	C23—C22—C27—C26	1.0 (4)
C8—C7—C12—C11	−2.6 (4)	S1—C22—C27—C26	179.5 (2)
C6—C7—C12—C11	179.2 (3)	C25—C26—C27—C22	−1.1 (4)
C11—C10—C13—F2′	73.8 (14)	C4—C5—N1—C1	58.2 (3)
C9—C10—C13—F2′	−107.6 (13)	C4—C5—N1—S1	−88.3 (3)
C11—C10—C13—F3	−89.8 (8)	C2—C1—N1—C5	−58.4 (3)
C9—C10—C13—F3	88.7 (8)	C2—C1—N1—S1	89.4 (2)
C11—C10—C13—F1′	−44.8 (11)	C5—N1—S1—O2	−154.37 (18)
C9—C10—C13—F1′	133.8 (11)	C1—N1—S1—O2	60.6 (2)
C11—C10—C13—F3′	−162.1 (10)	C5—N1—S1—O3	−25.8 (2)
C9—C10—C13—F3′	16.4 (10)	C1—N1—S1—O3	−170.82 (17)
C11—C10—C13—F1	32.2 (6)	C5—N1—S1—C22	91.6 (2)
C9—C10—C13—F1	−149.2 (6)	C1—N1—S1—C22	−53.5 (2)
C11—C10—C13—F2	145.6 (8)	C27—C22—S1—O2	161.2 (2)
C9—C10—C13—F2	−35.8 (9)	C23—C22—S1—O2	−20.3 (3)
C3—C4—C14—C15	−173.1 (3)	C27—C22—S1—O3	30.5 (3)
C5—C4—C14—C15	6.5 (5)	C23—C22—S1—O3	−151.0 (2)
C4—C14—C15—C16	−154.1 (3)	C27—C22—S1—N1	−85.1 (2)
C4—C14—C15—C20	30.7 (4)	C23—C22—S1—N1	93.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C26—H26···O1^{i}	0.95	2.58	3.458 (3)	154
C23—H23···F2′^{ii}	0.95	2.58	3.364 (12)	140
C23—H23···F1^{ii}	0.95	2.61	3.242 (7)	124
C1—H1*B*···O1^{iii}	0.99	2.46	3.293 (3)	142

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*.

(3*E*,5*E*)-1-(4-Nitrobenzenesulfonyl)-3,5-bis[4-(trifluoromethyl)benzylidene]piperidin-4-one (6)

Crystal data

$C_{27}H_{18}F_6N_2O_5S$
M_r = 596.49
Triclinic, $P\overline{1}$
a = 7.4346 (7) Å
b = 11.6714 (11) Å
c = 14.6479 (15) Å
α = 90.825 (5)°
β = 96.884 (5)°
γ = 98.971 (5)°
V = 1245.7 (2) Å^3
Z = 2
F(000) = 608
D_x = 1.590 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5944 reflections
θ = 2.3–26.4°

μ = 0.22 mm^{-1}
T = 150 K

Block, yellow
0.18 × 0.16 × 0.10 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: sealed tube
phi and ω scans
23092 measured reflections
4848 independent reflections

3350 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 26.0°, θ_{min} = 2.2°
h = −9→9
k = −14→14
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.129
S = 1.03
4848 reflections
370 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 1.0046P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.2697 (3)	0.2808 (2)	0.99958 (18)	0.0237 (6)
H1A	0.3550	0.3436	1.0356	0.028*
H1B	0.3403	0.2411	0.9594	0.028*
C2	0.1802 (3)	0.1958 (2)	1.06333 (18)	0.0218 (6)
C3	−0.0101 (3)	0.1329 (2)	1.03293 (18)	0.0228 (6)
C4	−0.0992 (4)	0.1547 (2)	0.93983 (18)	0.0233 (6)
C5	0.0042 (4)	0.2409 (2)	0.88225 (18)	0.0244 (6)
H5A	0.0792	0.2012	0.8444	0.029*
H5B	−0.0830	0.2778	0.8404	0.029*
C6	0.2579 (4)	0.1728 (2)	1.14696 (18)	0.0241 (6)
H6	0.1800	0.1231	1.1815	0.029*
C7	0.4435 (3)	0.2119 (2)	1.19402 (18)	0.0226 (6)
C8	0.4688 (4)	0.2154 (3)	1.29007 (19)	0.0310 (7)
H8	0.3655	0.1964	1.3225	0.037*
C9	0.6414 (4)	0.2460 (3)	1.3389 (2)	0.0319 (7)
H9	0.6561	0.2499	1.4043	0.038*
C10	0.7924 (4)	0.2710 (2)	1.29146 (19)	0.0266 (6)
C11	0.7714 (4)	0.2664 (2)	1.19627 (19)	0.0244 (6)
H11	0.8758	0.2830	1.1643	0.029*
C12	0.5980 (4)	0.2377 (2)	1.14770 (19)	0.0242 (6)
H12	0.5839	0.2354	1.0823	0.029*

C13	0.9797 (4)	0.2955 (3)	1.3438 (2)	0.0385 (8)
C14	−0.2679 (4)	0.0956 (2)	0.91185 (19)	0.0248 (6)
H14	−0.3218	0.0480	0.9566	0.030*
C15	−0.3797 (4)	0.0941 (2)	0.82299 (19)	0.0249 (6)
C16	−0.5702 (4)	0.0620 (2)	0.8190 (2)	0.0281 (6)
H16	−0.6225	0.0434	0.8741	0.034*
C17	−0.6826 (4)	0.0571 (2)	0.7373 (2)	0.0347 (7)
H17	−0.8116	0.0367	0.7362	0.042*
C18	−0.6080 (4)	0.0817 (3)	0.6561 (2)	0.0368 (7)
C19	−0.4201 (4)	0.1100 (3)	0.6575 (2)	0.0360 (7)
H19	−0.3687	0.1257	0.6018	0.043*
C20	−0.3071 (4)	0.1154 (2)	0.7399 (2)	0.0298 (6)
H20	−0.1780	0.1339	0.7402	0.036*
C21	−0.7317 (5)	0.0732 (3)	0.5674 (3)	0.0512 (9)
C22	0.2910 (3)	0.4401 (2)	0.80587 (18)	0.0225 (6)
C23	0.4795 (4)	0.4413 (2)	0.82156 (19)	0.0268 (6)
H23	0.5426	0.4528	0.8821	0.032*
C24	0.5737 (4)	0.4254 (2)	0.7475 (2)	0.0317 (7)
H24	0.7028	0.4268	0.7562	0.038*
C25	0.4765 (4)	0.4076 (2)	0.6611 (2)	0.0299 (7)
C26	0.2900 (4)	0.4064 (2)	0.6445 (2)	0.0323 (7)
H26	0.2278	0.3947	0.5838	0.039*
C27	0.1952 (4)	0.4224 (2)	0.71799 (19)	0.0287 (6)
H27	0.0663	0.4214	0.7086	0.034*
F1	1.1028 (2)	0.35879 (16)	1.29868 (14)	0.0502 (5)
F2	1.0477 (2)	0.19669 (19)	1.36152 (16)	0.0626 (7)
F3	0.9829 (3)	0.3491 (3)	1.42395 (16)	0.0943 (10)
F4	−0.6494 (4)	0.1212 (3)	0.49823 (18)	0.1085 (12)
F5	−0.8768 (4)	0.1228 (3)	0.5723 (2)	0.1092 (11)
F6	−0.7900 (3)	−0.03590 (19)	0.53857 (15)	0.0697 (7)
N1	0.1234 (3)	0.32961 (18)	0.94353 (15)	0.0233 (5)
N2	0.5775 (4)	0.3884 (2)	0.5824 (2)	0.0431 (7)
O1	−0.0883 (2)	0.06280 (16)	1.08281 (13)	0.0290 (5)
O2	0.2915 (2)	0.52922 (15)	0.96959 (12)	0.0265 (4)
O3	−0.0020 (2)	0.48987 (16)	0.86577 (13)	0.0277 (4)
O4	0.7417 (3)	0.3840 (2)	0.59848 (18)	0.0606 (7)
O5	0.4897 (4)	0.3747 (2)	0.50595 (18)	0.0665 (8)
S1	0.16976 (9)	0.45813 (6)	0.90097 (5)	0.02288 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0206 (13)	0.0262 (14)	0.0226 (14)	0.0013 (11)	−0.0016 (11)	0.0016 (11)
C2	0.0223 (13)	0.0209 (13)	0.0217 (14)	0.0014 (10)	0.0033 (11)	0.0011 (11)
C3	0.0226 (14)	0.0251 (14)	0.0213 (14)	0.0033 (11)	0.0058 (11)	−0.0011 (11)
C4	0.0231 (14)	0.0232 (14)	0.0223 (14)	0.0019 (11)	0.0000 (11)	−0.0008 (11)
C5	0.0227 (14)	0.0261 (14)	0.0209 (14)	−0.0019 (11)	−0.0037 (11)	−0.0004 (11)
C6	0.0226 (14)	0.0263 (14)	0.0241 (15)	0.0032 (11)	0.0060 (12)	0.0022 (11)

C7	0.0207 (13)	0.0231 (13)	0.0241 (14)	0.0049 (11)	0.0005 (11)	0.0050 (11)
C8	0.0251 (15)	0.0452 (17)	0.0244 (16)	0.0095 (13)	0.0035 (12)	0.0077 (13)
C9	0.0290 (15)	0.0486 (18)	0.0200 (15)	0.0157 (13)	−0.0015 (12)	0.0022 (13)
C10	0.0250 (14)	0.0279 (15)	0.0275 (15)	0.0096 (11)	−0.0016 (12)	0.0009 (12)
C11	0.0215 (14)	0.0253 (14)	0.0273 (15)	0.0054 (11)	0.0041 (12)	0.0012 (11)
C12	0.0270 (14)	0.0235 (14)	0.0226 (14)	0.0046 (11)	0.0043 (12)	0.0010 (11)
C13	0.0276 (16)	0.056 (2)	0.0327 (18)	0.0130 (15)	−0.0020 (14)	0.0042 (15)
C14	0.0260 (14)	0.0225 (14)	0.0255 (15)	0.0025 (11)	0.0035 (12)	−0.0008 (11)
C15	0.0212 (14)	0.0195 (13)	0.0309 (16)	−0.0018 (11)	−0.0027 (12)	−0.0011 (11)
C16	0.0238 (14)	0.0237 (14)	0.0354 (17)	0.0001 (11)	0.0025 (13)	0.0005 (12)
C17	0.0231 (15)	0.0285 (16)	0.048 (2)	0.0009 (12)	−0.0087 (14)	−0.0014 (14)
C18	0.0375 (17)	0.0289 (16)	0.0375 (18)	0.0003 (13)	−0.0162 (14)	0.0029 (13)
C19	0.0390 (17)	0.0338 (16)	0.0294 (17)	−0.0060 (13)	−0.0039 (14)	0.0019 (13)
C20	0.0234 (14)	0.0318 (15)	0.0306 (16)	−0.0035 (12)	−0.0007 (12)	−0.0010 (12)
C21	0.049 (2)	0.044 (2)	0.050 (2)	−0.0060 (17)	−0.0198 (18)	0.0045 (17)
C22	0.0234 (14)	0.0209 (13)	0.0223 (14)	0.0018 (11)	0.0016 (11)	0.0019 (11)
C23	0.0269 (15)	0.0294 (15)	0.0226 (15)	0.0007 (11)	0.0017 (12)	0.0012 (11)
C24	0.0271 (15)	0.0328 (16)	0.0363 (18)	0.0060 (12)	0.0069 (13)	0.0028 (13)
C25	0.0362 (16)	0.0265 (15)	0.0289 (16)	0.0049 (12)	0.0124 (13)	−0.0017 (12)
C26	0.0381 (17)	0.0363 (16)	0.0214 (15)	0.0044 (13)	0.0014 (13)	0.0008 (12)
C27	0.0246 (14)	0.0341 (16)	0.0270 (16)	0.0053 (12)	0.0001 (12)	0.0028 (12)
F1	0.0271 (10)	0.0541 (12)	0.0628 (13)	−0.0043 (8)	−0.0087 (9)	0.0168 (10)
F2	0.0273 (10)	0.0755 (15)	0.0861 (17)	0.0151 (10)	−0.0017 (10)	0.0436 (13)
F3	0.0377 (12)	0.185 (3)	0.0528 (15)	0.0176 (15)	−0.0155 (11)	−0.0596 (17)
F4	0.095 (2)	0.135 (2)	0.0626 (17)	−0.0474 (18)	−0.0445 (15)	0.0581 (17)
F5	0.090 (2)	0.134 (3)	0.099 (2)	0.0623 (19)	−0.0620 (17)	−0.0291 (18)
F6	0.0825 (16)	0.0577 (14)	0.0514 (14)	−0.0123 (12)	−0.0309 (12)	−0.0028 (10)
N1	0.0222 (12)	0.0226 (12)	0.0227 (12)	−0.0013 (9)	−0.0014 (10)	0.0052 (9)
N2	0.0588 (19)	0.0370 (15)	0.0371 (17)	0.0057 (13)	0.0235 (15)	−0.0053 (12)
O1	0.0271 (10)	0.0322 (11)	0.0261 (11)	−0.0026 (8)	0.0056 (9)	0.0045 (8)
O2	0.0298 (10)	0.0256 (10)	0.0217 (10)	−0.0017 (8)	0.0016 (8)	−0.0017 (8)
O3	0.0228 (10)	0.0318 (11)	0.0290 (11)	0.0072 (8)	0.0012 (8)	0.0060 (8)
O4	0.0484 (16)	0.0762 (19)	0.0636 (18)	0.0120 (13)	0.0315 (14)	−0.0027 (14)
O5	0.083 (2)	0.088 (2)	0.0329 (15)	0.0161 (16)	0.0222 (15)	−0.0119 (14)
S1	0.0228 (3)	0.0246 (3)	0.0204 (4)	0.0017 (3)	0.0020 (3)	0.0024 (3)

Geometric parameters (Å, º)

C1—N1	1.474 (3)	C15—C20	1.400 (4)
C1—C2	1.507 (3)	C15—C16	1.402 (4)
C1—H1A	0.9900	C16—C17	1.370 (4)
C1—H1B	0.9900	C16—H16	0.9500
C2—C6	1.339 (4)	C17—C18	1.386 (4)
C2—C3	1.501 (4)	C17—H17	0.9500
C3—O1	1.230 (3)	C18—C19	1.383 (4)
C3—C4	1.486 (4)	C18—C21	1.491 (4)
C4—C14	1.348 (4)	C19—C20	1.379 (4)
C4—C5	1.502 (4)	C19—H19	0.9500

C5—N1	1.469 (3)	C20—H20	0.9500
C5—H5A	0.9900	C21—F5	1.310 (4)
C5—H5B	0.9900	C21—F6	1.325 (4)
C6—C7	1.468 (4)	C21—F4	1.331 (4)
C6—H6	0.9500	C22—C23	1.390 (4)
C7—C8	1.396 (4)	C22—C27	1.391 (4)
C7—C12	1.398 (4)	C22—S1	1.775 (3)
C8—C9	1.384 (4)	C23—C24	1.385 (4)
C8—H8	0.9500	C23—H23	0.9500
C9—C10	1.385 (4)	C24—C25	1.376 (4)
C9—H9	0.9500	C24—H24	0.9500
C10—C11	1.384 (4)	C25—C26	1.376 (4)
C10—C13	1.491 (4)	C25—N2	1.481 (4)
C11—C12	1.385 (4)	C26—C27	1.381 (4)
C11—H11	0.9500	C26—H26	0.9500
C12—H12	0.9500	C27—H27	0.9500
C13—F3	1.318 (4)	N1—S1	1.635 (2)
C13—F1	1.329 (4)	N2—O5	1.222 (4)
C13—F2	1.347 (4)	N2—O4	1.224 (4)
C14—C15	1.456 (4)	O2—S1	1.4290 (19)
C14—H14	0.9500	O3—S1	1.4246 (19)

N1—C1—C2	107.8 (2)	C16—C15—C14	118.5 (3)
N1—C1—H1A	110.1	C17—C16—C15	121.3 (3)
C2—C1—H1A	110.1	C17—C16—H16	119.4
N1—C1—H1B	110.1	C15—C16—H16	119.4
C2—C1—H1B	110.1	C16—C17—C18	120.0 (3)
H1A—C1—H1B	108.5	C16—C17—H17	120.0
C6—C2—C3	116.9 (2)	C18—C17—H17	120.0
C6—C2—C1	124.4 (2)	C19—C18—C17	120.0 (3)
C3—C2—C1	118.7 (2)	C19—C18—C21	120.6 (3)
O1—C3—C4	120.8 (2)	C17—C18—C21	119.4 (3)
O1—C3—C2	120.8 (2)	C20—C19—C18	120.0 (3)
C4—C3—C2	118.3 (2)	C20—C19—H19	120.0
C14—C4—C3	118.1 (2)	C18—C19—H19	120.0
C14—C4—C5	123.8 (2)	C19—C20—C15	121.1 (3)
C3—C4—C5	118.1 (2)	C19—C20—H20	119.5
N1—C5—C4	108.8 (2)	C15—C20—H20	119.5
N1—C5—H5A	109.9	F5—C21—F6	107.5 (3)
C4—C5—H5A	109.9	F5—C21—F4	106.6 (3)
N1—C5—H5B	109.9	F6—C21—F4	104.0 (3)
C4—C5—H5B	109.9	F5—C21—C18	112.8 (3)
H5A—C5—H5B	108.3	F6—C21—C18	112.1 (3)
C2—C6—C7	130.6 (2)	F4—C21—C18	113.2 (3)
C2—C6—H6	114.7	C23—C22—C27	121.6 (3)
C7—C6—H6	114.7	C23—C22—S1	119.0 (2)
C8—C7—C12	118.4 (2)	C27—C22—S1	119.4 (2)
C8—C7—C6	118.2 (2)	C24—C23—C22	119.0 (3)

C12—C7—C6	123.3 (2)	C24—C23—H23	120.5
C9—C8—C7	121.3 (3)	C22—C23—H23	120.5
C9—C8—H8	119.4	C25—C24—C23	118.6 (3)
C7—C8—H8	119.4	C25—C24—H24	120.7
C8—C9—C10	119.3 (3)	C23—C24—H24	120.7
C8—C9—H9	120.4	C26—C25—C24	123.0 (3)
C10—C9—H9	120.4	C26—C25—N2	118.6 (3)
C11—C10—C9	120.6 (3)	C24—C25—N2	118.4 (3)
C11—C10—C13	119.9 (2)	C25—C26—C27	118.7 (3)
C9—C10—C13	119.4 (3)	C25—C26—H26	120.7
C10—C11—C12	119.9 (2)	C27—C26—H26	120.7
C10—C11—H11	120.0	C26—C27—C22	119.1 (3)
C12—C11—H11	120.0	C26—C27—H27	120.5
C11—C12—C7	120.6 (2)	C22—C27—H27	120.5
C11—C12—H12	119.7	C5—N1—C1	111.8 (2)
C7—C12—H12	119.7	C5—N1—S1	116.02 (17)
F3—C13—F1	107.2 (3)	C1—N1—S1	119.59 (17)
F3—C13—F2	106.5 (3)	O5—N2—O4	124.4 (3)
F1—C13—F2	104.8 (2)	O5—N2—C25	117.5 (3)
F3—C13—C10	112.9 (3)	O4—N2—C25	118.1 (3)
F1—C13—C10	113.8 (2)	O3—S1—O2	120.83 (12)
F2—C13—C10	111.1 (3)	O3—S1—N1	106.37 (11)
C4—C14—C15	129.4 (3)	O2—S1—N1	107.05 (11)
C4—C14—H14	115.3	O3—S1—C22	107.42 (12)
C15—C14—H14	115.3	O2—S1—C22	107.99 (12)
C20—C15—C16	117.6 (3)	N1—S1—C22	106.35 (12)
C20—C15—C14	123.8 (2)		

N1—C1—C2—C6	−149.6 (3)	C17—C18—C19—C20	−1.0 (4)
N1—C1—C2—C3	29.4 (3)	C21—C18—C19—C20	−178.8 (3)
C6—C2—C3—O1	−0.8 (4)	C18—C19—C20—C15	−0.8 (4)
C1—C2—C3—O1	−180.0 (2)	C16—C15—C20—C19	2.8 (4)
C6—C2—C3—C4	−178.9 (2)	C14—C15—C20—C19	178.4 (3)
C1—C2—C3—C4	2.0 (4)	C19—C18—C21—F5	−136.3 (3)
O1—C3—C4—C14	0.1 (4)	C17—C18—C21—F5	45.9 (4)
C2—C3—C4—C14	178.2 (2)	C19—C18—C21—F6	102.2 (4)
O1—C3—C4—C5	−179.7 (2)	C17—C18—C21—F6	−75.6 (4)
C2—C3—C4—C5	−1.6 (4)	C19—C18—C21—F4	−15.1 (5)
C14—C4—C5—N1	149.9 (3)	C17—C18—C21—F4	167.1 (3)
C3—C4—C5—N1	−30.3 (3)	C27—C22—C23—C24	0.5 (4)
C3—C2—C6—C7	174.9 (2)	S1—C22—C23—C24	179.0 (2)
C1—C2—C6—C7	−6.0 (5)	C22—C23—C24—C25	−0.8 (4)
C2—C6—C7—C8	154.6 (3)	C23—C24—C25—C26	0.9 (4)
C2—C6—C7—C12	−30.2 (4)	C23—C24—C25—N2	−178.9 (2)
C12—C7—C8—C9	1.4 (4)	C24—C25—C26—C27	−0.8 (4)
C6—C7—C8—C9	176.9 (3)	N2—C25—C26—C27	179.0 (2)
C7—C8—C9—C10	−1.5 (4)	C25—C26—C27—C22	0.5 (4)
C8—C9—C10—C11	0.5 (4)	C23—C22—C27—C26	−0.4 (4)

C8—C9—C10—C13	−175.5 (3)	S1—C22—C27—C26	−178.8 (2)
C9—C10—C11—C12	0.6 (4)	C4—C5—N1—C1	66.3 (3)
C13—C10—C11—C12	176.6 (2)	C4—C5—N1—S1	−151.72 (18)
C10—C11—C12—C7	−0.7 (4)	C2—C1—N1—C5	−65.4 (3)
C8—C7—C12—C11	−0.3 (4)	C2—C1—N1—S1	154.18 (18)
C6—C7—C12—C11	−175.5 (2)	C26—C25—N2—O5	1.4 (4)
C11—C10—C13—F3	150.4 (3)	C24—C25—N2—O5	−178.8 (3)
C9—C10—C13—F3	−33.6 (4)	C26—C25—N2—O4	−176.5 (3)
C11—C10—C13—F1	27.9 (4)	C24—C25—N2—O4	3.3 (4)
C9—C10—C13—F1	−156.0 (3)	C5—N1—S1—O3	51.6 (2)
C11—C10—C13—F2	−90.1 (3)	C1—N1—S1—O3	−169.49 (19)
C9—C10—C13—F2	86.0 (3)	C5—N1—S1—O2	−177.90 (18)
C3—C4—C14—C15	−175.3 (3)	C1—N1—S1—O2	−39.0 (2)
C5—C4—C14—C15	4.5 (5)	C5—N1—S1—C22	−62.6 (2)
C4—C14—C15—C20	27.5 (5)	C1—N1—S1—C22	76.2 (2)
C4—C14—C15—C16	−157.0 (3)	C23—C22—S1—O3	162.1 (2)
C20—C15—C16—C17	−3.1 (4)	C27—C22—S1—O3	−19.4 (2)
C14—C15—C16—C17	−178.9 (3)	C23—C22—S1—O2	30.3 (2)
C15—C16—C17—C18	1.3 (4)	C27—C22—S1—O2	−151.2 (2)
C16—C17—C18—C19	0.7 (4)	C23—C22—S1—N1	−84.4 (2)
C16—C17—C18—C21	178.6 (3)	C27—C22—S1—N1	94.1 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C24—H24···O3^{i}	0.95	2.56	3.380 (4)	145
C23—H23···O2^{ii}	0.95	2.36	3.304 (3)	176

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*+2.

(3*E*,5*E*)-1-(4-Cyanobenzenesulfonyl)-3,5-bis[4-(trifluoromethyl)benzylidene]piperidin-4-one dichloromethane monosolvate (7)

Crystal data

$C_{28}H_{18}F_6N_2O_3S{\cdot}CH_2Cl_2$
M_r = 661.43
Triclinic, $P\overline{1}$
a = 9.7338 (6) Å
b = 12.4558 (9) Å
c = 12.6397 (9) Å
α = 70.586 (3)°
β = 88.379 (4)°
γ = 81.726 (3)°
V = 1429.97 (17) Å^3

Z = 2
F(000) = 672
D_x = 1.536 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5958 reflections
θ = 2.6–26.5°
μ = 0.38 mm^{-1}
T = 150 K
Block, yellow
0.20 × 0.15 × 0.11 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: sealed tube
phi and *ω* scans

24790 measured reflections
5604 independent reflections
3869 reflections with $I > 2\sigma(I)$
R_{int} = 0.070

θ_{max} = 26.0°, θ_{min} = 2.6°
h = −12→12
k = −15→15
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.109
S = 1.00
5604 reflections
388 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.7221P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.2251 (3)	0.6874 (2)	0.3737 (2)	0.0239 (6)
H1A	0.3181	0.7010	0.3429	0.029*
H1B	0.1587	0.7048	0.3104	0.029*
C2	0.2295 (2)	0.5639 (2)	0.4483 (2)	0.0203 (5)
C3	0.1347 (3)	0.5379 (2)	0.5467 (2)	0.0228 (6)
C4	0.0349 (2)	0.6341 (2)	0.5613 (2)	0.0203 (5)
C5	0.0378 (2)	0.7528 (2)	0.4781 (2)	0.0221 (5)
H5A	−0.0243	0.7657	0.4131	0.027*
H5B	0.0057	0.8116	0.5136	0.027*
C6	0.3168 (2)	0.4754 (2)	0.4360 (2)	0.0222 (6)
H6	0.3107	0.4028	0.4912	0.027*
C7	0.4201 (2)	0.4745 (2)	0.3497 (2)	0.0214 (5)
C8	0.5350 (3)	0.3884 (2)	0.3780 (2)	0.0239 (6)
H8	0.5408	0.3317	0.4505	0.029*
C9	0.6399 (3)	0.3837 (2)	0.3037 (2)	0.0249 (6)
H9	0.7181	0.3254	0.3255	0.030*
C10	0.6306 (3)	0.4647 (2)	0.1966 (2)	0.0230 (6)
C11	0.5137 (3)	0.5474 (2)	0.1649 (2)	0.0253 (6)
H11	0.5054	0.6010	0.0908	0.030*
C12	0.4095 (3)	0.5524 (2)	0.2400 (2)	0.0238 (6)
H12	0.3300	0.6092	0.2172	0.029*
C13	0.7436 (3)	0.4651 (2)	0.1152 (2)	0.0306 (6)
C14	−0.0505 (3)	0.6104 (2)	0.6484 (2)	0.0238 (6)
H14	−0.0352	0.5338	0.6993	0.029*
C15	−0.1642 (2)	0.6869 (2)	0.6756 (2)	0.0232 (6)
C16	−0.2007 (3)	0.6650 (2)	0.7880 (2)	0.0318 (6)
H16	−0.1463	0.6056	0.8451	0.038*
C17	−0.3144 (3)	0.7282 (3)	0.8170 (2)	0.0348 (7)

H17	−0.3365	0.7136	0.8937	0.042*
C18	−0.3965 (3)	0.8132 (2)	0.7342 (2)	0.0278 (6)
C19	−0.3622 (3)	0.8373 (2)	0.6225 (2)	0.0275 (6)
H19	−0.4180	0.8960	0.5658	0.033*
C20	−0.2459 (2)	0.7755 (2)	0.5939 (2)	0.0246 (6)
H20	−0.2213	0.7936	0.5174	0.030*
C21	−0.5244 (3)	0.8746 (3)	0.7680 (3)	0.0353 (7)
C22	0.1465 (2)	0.9772 (2)	0.2868 (2)	0.0199 (5)
C23	0.1993 (3)	0.9807 (2)	0.1829 (2)	0.0257 (6)
H23	0.2846	0.9350	0.1780	0.031*
C24	0.1271 (3)	1.0512 (2)	0.0868 (2)	0.0319 (6)
H24	0.1629	1.0550	0.0152	0.038*
C25	0.0013 (3)	1.1169 (2)	0.0953 (2)	0.0294 (6)
C26	−0.0518 (3)	1.1122 (2)	0.1996 (2)	0.0303 (6)
H26	−0.1380	1.1568	0.2048	0.036*
C27	0.0212 (3)	1.0423 (2)	0.2959 (2)	0.0264 (6)
H27	−0.0140	1.0388	0.3676	0.032*
C28	−0.0742 (3)	1.1910 (3)	−0.0048 (3)	0.0447 (8)
C29	0.3476 (3)	0.2120 (3)	0.7732 (2)	0.0421 (8)
H29A	0.4133	0.1976	0.7166	0.050*
H29B	0.3169	0.2960	0.7507	0.050*
Cl1	0.20281 (8)	0.14143 (7)	0.77593 (7)	0.0483 (2)
Cl2	0.43281 (8)	0.16459 (7)	0.90498 (7)	0.0445 (2)
F1	0.80484 (18)	0.56012 (16)	0.08932 (16)	0.0527 (5)
F2	0.69830 (18)	0.46293 (18)	0.01714 (14)	0.0535 (5)
F3	0.84560 (17)	0.37668 (15)	0.15218 (14)	0.0473 (5)
F4	−0.60578 (18)	0.9403 (2)	0.68140 (16)	0.0634 (6)
F5	−0.49708 (16)	0.94232 (15)	0.82446 (15)	0.0434 (4)
F6	−0.60193 (18)	0.80030 (17)	0.83699 (19)	0.0636 (6)
N1	0.1814 (2)	0.76125 (17)	0.44130 (16)	0.0208 (5)
N2	−0.1347 (3)	1.2483 (3)	−0.0848 (2)	0.0687 (10)
O1	0.1362 (2)	0.43929 (15)	0.61050 (15)	0.0326 (5)
O2	0.38056 (16)	0.86676 (15)	0.38543 (14)	0.0261 (4)
O3	0.19401 (18)	0.92877 (15)	0.49955 (14)	0.0278 (4)
S1	0.23658 (6)	0.88541 (5)	0.41058 (5)	0.02072 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0293 (14)	0.0212 (14)	0.0206 (13)	−0.0014 (11)	0.0024 (11)	−0.0073 (11)
C2	0.0221 (12)	0.0201 (13)	0.0180 (12)	−0.0031 (10)	−0.0042 (10)	−0.0048 (10)
C3	0.0272 (14)	0.0213 (14)	0.0206 (13)	−0.0073 (11)	−0.0010 (11)	−0.0059 (11)
C4	0.0225 (13)	0.0215 (13)	0.0174 (12)	−0.0061 (10)	−0.0003 (10)	−0.0058 (11)
C5	0.0219 (13)	0.0204 (13)	0.0210 (13)	−0.0039 (10)	0.0017 (10)	−0.0027 (11)
C6	0.0262 (13)	0.0190 (13)	0.0201 (13)	−0.0050 (11)	−0.0051 (10)	−0.0035 (11)
C7	0.0247 (13)	0.0189 (13)	0.0220 (13)	−0.0053 (11)	−0.0042 (10)	−0.0073 (11)
C8	0.0275 (14)	0.0222 (14)	0.0199 (13)	−0.0037 (11)	−0.0038 (11)	−0.0036 (11)
C9	0.0245 (13)	0.0231 (14)	0.0251 (14)	0.0003 (11)	−0.0047 (11)	−0.0066 (11)

C10	0.0255 (13)	0.0227 (14)	0.0229 (13)	−0.0055 (11)	−0.0027 (11)	−0.0091 (11)
C11	0.0314 (14)	0.0225 (14)	0.0192 (13)	−0.0015 (11)	−0.0018 (11)	−0.0039 (11)
C12	0.0251 (13)	0.0209 (14)	0.0225 (13)	0.0025 (11)	−0.0050 (11)	−0.0054 (11)
C13	0.0306 (15)	0.0306 (16)	0.0286 (15)	−0.0009 (12)	−0.0007 (12)	−0.0086 (13)
C14	0.0285 (13)	0.0197 (14)	0.0222 (13)	−0.0059 (11)	−0.0011 (11)	−0.0042 (11)
C15	0.0234 (13)	0.0230 (14)	0.0254 (14)	−0.0096 (11)	0.0033 (11)	−0.0086 (11)
C16	0.0301 (15)	0.0357 (17)	0.0250 (15)	−0.0018 (12)	0.0021 (12)	−0.0054 (13)
C17	0.0306 (15)	0.0457 (19)	0.0275 (15)	−0.0038 (14)	0.0050 (12)	−0.0125 (14)
C18	0.0224 (13)	0.0310 (16)	0.0363 (16)	−0.0080 (12)	0.0024 (12)	−0.0179 (13)
C19	0.0251 (14)	0.0252 (15)	0.0328 (15)	−0.0066 (11)	−0.0018 (12)	−0.0089 (12)
C20	0.0255 (14)	0.0252 (14)	0.0237 (14)	−0.0105 (11)	0.0030 (11)	−0.0062 (11)
C21	0.0297 (15)	0.0407 (18)	0.0408 (17)	−0.0069 (13)	0.0039 (13)	−0.0198 (15)
C22	0.0196 (12)	0.0157 (13)	0.0228 (13)	−0.0040 (10)	−0.0013 (10)	−0.0034 (10)
C23	0.0245 (13)	0.0257 (15)	0.0233 (14)	0.0000 (11)	0.0008 (11)	−0.0049 (12)
C24	0.0366 (16)	0.0353 (17)	0.0202 (14)	−0.0016 (13)	−0.0018 (12)	−0.0058 (12)
C25	0.0319 (15)	0.0248 (15)	0.0285 (15)	0.0014 (12)	−0.0098 (12)	−0.0065 (12)
C26	0.0242 (14)	0.0263 (15)	0.0385 (17)	0.0034 (11)	−0.0038 (12)	−0.0109 (13)
C27	0.0283 (14)	0.0240 (14)	0.0257 (14)	−0.0026 (11)	0.0040 (11)	−0.0071 (12)
C28	0.051 (2)	0.0412 (19)	0.0376 (18)	0.0107 (15)	−0.0149 (15)	−0.0138 (16)
C29	0.0449 (18)	0.0406 (19)	0.0347 (17)	−0.0071 (15)	−0.0006 (14)	−0.0040 (15)
Cl1	0.0446 (4)	0.0553 (5)	0.0397 (4)	−0.0100 (4)	−0.0125 (3)	−0.0065 (4)
Cl2	0.0505 (5)	0.0391 (5)	0.0454 (5)	−0.0031 (4)	−0.0147 (4)	−0.0159 (4)
F1	0.0508 (11)	0.0464 (12)	0.0633 (13)	−0.0214 (9)	0.0239 (9)	−0.0172 (10)
F2	0.0449 (10)	0.0923 (16)	0.0309 (9)	−0.0074 (10)	0.0034 (8)	−0.0314 (10)
F3	0.0391 (10)	0.0484 (11)	0.0426 (10)	0.0122 (8)	0.0094 (8)	−0.0077 (9)
F4	0.0376 (10)	0.0985 (17)	0.0558 (12)	0.0263 (10)	−0.0150 (9)	−0.0415 (12)
F5	0.0393 (10)	0.0466 (11)	0.0543 (11)	−0.0010 (8)	0.0011 (8)	−0.0318 (9)
F6	0.0409 (10)	0.0520 (13)	0.1059 (17)	−0.0171 (9)	0.0368 (11)	−0.0352 (12)
N1	0.0243 (11)	0.0163 (11)	0.0204 (11)	−0.0036 (9)	0.0016 (9)	−0.0042 (9)
N2	0.085 (2)	0.066 (2)	0.0404 (17)	0.0332 (18)	−0.0275 (17)	−0.0141 (16)
O1	0.0449 (12)	0.0198 (10)	0.0270 (10)	−0.0033 (8)	0.0094 (9)	−0.0011 (8)
O2	0.0189 (9)	0.0283 (10)	0.0272 (10)	−0.0040 (8)	−0.0005 (7)	−0.0036 (8)
O3	0.0376 (10)	0.0269 (10)	0.0216 (9)	−0.0077 (8)	0.0009 (8)	−0.0102 (8)
S1	0.0228 (3)	0.0200 (3)	0.0176 (3)	−0.0044 (2)	−0.0008 (2)	−0.0033 (3)

Geometric parameters (Å, º)

C1—N1	1.465 (3)	C16—C17	1.378 (4)
C1—C2	1.508 (3)	C16—H16	0.9500
C1—H1A	0.9900	C17—C18	1.386 (4)
C1—H1B	0.9900	C17—H17	0.9500
C2—C6	1.341 (3)	C18—C19	1.384 (4)
C2—C3	1.502 (3)	C18—C21	1.494 (4)
C3—O1	1.223 (3)	C19—C20	1.384 (4)
C3—C4	1.488 (3)	C19—H19	0.9500
C4—C14	1.340 (3)	C20—H20	0.9500
C4—C5	1.506 (3)	C21—F4	1.328 (3)
C5—N1	1.468 (3)	C21—F5	1.329 (3)

C5—H5A	0.9900	C21—F6	1.347 (3)
C5—H5B	0.9900	C22—C23	1.385 (3)
C6—C7	1.464 (3)	C22—C27	1.388 (3)
C6—H6	0.9500	C22—S1	1.768 (2)
C7—C8	1.396 (3)	C23—C24	1.379 (3)
C7—C12	1.400 (3)	C23—H23	0.9500
C8—C9	1.376 (3)	C24—C25	1.394 (4)
C8—H8	0.9500	C24—H24	0.9500
C9—C10	1.390 (3)	C25—C26	1.387 (4)
C9—H9	0.9500	C25—C28	1.440 (4)
C10—C11	1.390 (3)	C26—C27	1.383 (4)
C10—C13	1.484 (4)	C26—H26	0.9500
C11—C12	1.378 (3)	C27—H27	0.9500
C11—H11	0.9500	C28—N2	1.145 (4)
C12—H12	0.9500	C29—Cl2	1.759 (3)
C13—F2	1.337 (3)	C29—Cl1	1.759 (3)
C13—F3	1.337 (3)	C29—H29A	0.9900
C13—F1	1.341 (3)	C29—H29B	0.9900
C14—C15	1.464 (4)	N1—S1	1.631 (2)
C14—H14	0.9500	O2—S1	1.4315 (17)
C15—C20	1.398 (3)	O3—S1	1.4286 (18)
C15—C16	1.400 (4)		

N1—C1—C2	108.13 (19)	C17—C16—H16	119.5
N1—C1—H1A	110.1	C15—C16—H16	119.5
C2—C1—H1A	110.1	C16—C17—C18	120.0 (3)
N1—C1—H1B	110.1	C16—C17—H17	120.0
C2—C1—H1B	110.1	C18—C17—H17	120.0
H1A—C1—H1B	108.4	C19—C18—C17	120.2 (2)
C6—C2—C3	117.1 (2)	C19—C18—C21	121.2 (3)
C6—C2—C1	124.5 (2)	C17—C18—C21	118.6 (2)
C3—C2—C1	118.3 (2)	C20—C19—C18	119.6 (2)
O1—C3—C4	121.1 (2)	C20—C19—H19	120.2
O1—C3—C2	120.6 (2)	C18—C19—H19	120.2
C4—C3—C2	118.3 (2)	C19—C20—C15	121.2 (2)
C14—C4—C3	118.1 (2)	C19—C20—H20	119.4
C14—C4—C5	123.9 (2)	C15—C20—H20	119.4
C3—C4—C5	117.9 (2)	F4—C21—F5	106.6 (2)
N1—C5—C4	107.7 (2)	F4—C21—F6	106.9 (2)
N1—C5—H5A	110.2	F5—C21—F6	105.2 (2)
C4—C5—H5A	110.2	F4—C21—C18	113.1 (2)
N1—C5—H5B	110.2	F5—C21—C18	113.0 (2)
C4—C5—H5B	110.2	F6—C21—C18	111.5 (2)
H5A—C5—H5B	108.5	C23—C22—C27	121.2 (2)
C2—C6—C7	129.4 (2)	C23—C22—S1	119.91 (19)
C2—C6—H6	115.3	C27—C22—S1	118.91 (19)
C7—C6—H6	115.3	C24—C23—C22	119.5 (2)
C8—C7—C12	117.9 (2)	C24—C23—H23	120.3

C8—C7—C6	117.8 (2)	C22—C23—H23	120.3
C12—C7—C6	124.3 (2)	C23—C24—C25	119.7 (3)
C9—C8—C7	121.7 (2)	C23—C24—H24	120.2
C9—C8—H8	119.2	C25—C24—H24	120.2
C7—C8—H8	119.2	C26—C25—C24	120.6 (2)
C8—C9—C10	119.6 (2)	C26—C25—C28	119.5 (3)
C8—C9—H9	120.2	C24—C25—C28	119.9 (3)
C10—C9—H9	120.2	C27—C26—C25	119.7 (2)
C11—C10—C9	119.5 (2)	C27—C26—H26	120.2
C11—C10—C13	119.2 (2)	C25—C26—H26	120.2
C9—C10—C13	121.3 (2)	C26—C27—C22	119.4 (2)
C12—C11—C10	120.6 (2)	C26—C27—H27	120.3
C12—C11—H11	119.7	C22—C27—H27	120.3
C10—C11—H11	119.7	N2—C28—C25	178.9 (4)
C11—C12—C7	120.5 (2)	Cl2—C29—Cl1	111.56 (16)
C11—C12—H12	119.8	Cl2—C29—H29A	109.3
C7—C12—H12	119.8	Cl1—C29—H29A	109.3
F2—C13—F3	106.7 (2)	Cl2—C29—H29B	109.3
F2—C13—F1	105.2 (2)	Cl1—C29—H29B	109.3
F3—C13—F1	105.8 (2)	H29A—C29—H29B	108.0
F2—C13—C10	112.8 (2)	C1—N1—C5	111.55 (19)
F3—C13—C10	113.6 (2)	C1—N1—S1	119.52 (16)
F1—C13—C10	112.2 (2)	C5—N1—S1	118.39 (16)
C4—C14—C15	128.6 (2)	O3—S1—O2	120.45 (11)
C4—C14—H14	115.7	O3—S1—N1	106.35 (10)
C15—C14—H14	115.7	O2—S1—N1	106.35 (11)
C20—C15—C16	117.9 (2)	O3—S1—C22	107.96 (11)
C20—C15—C14	123.1 (2)	O2—S1—C22	108.23 (11)
C16—C15—C14	118.8 (2)	N1—S1—C22	106.76 (11)
C17—C16—C15	121.0 (3)		

N1—C1—C2—C6	−149.1 (2)	C16—C17—C18—C19	−2.1 (4)
N1—C1—C2—C3	27.8 (3)	C16—C17—C18—C21	175.9 (3)
C6—C2—C3—O1	−1.6 (4)	C17—C18—C19—C20	0.5 (4)
C1—C2—C3—O1	−178.8 (2)	C21—C18—C19—C20	−177.4 (2)
C6—C2—C3—C4	−178.9 (2)	C18—C19—C20—C15	1.6 (4)
C1—C2—C3—C4	3.9 (3)	C16—C15—C20—C19	−2.1 (4)
O1—C3—C4—C14	2.0 (4)	C14—C15—C20—C19	172.5 (2)
C2—C3—C4—C14	179.3 (2)	C19—C18—C21—F4	8.1 (4)
O1—C3—C4—C5	−179.0 (2)	C17—C18—C21—F4	−169.8 (3)
C2—C3—C4—C5	−1.7 (3)	C19—C18—C21—F5	−113.1 (3)
C14—C4—C5—N1	147.1 (2)	C17—C18—C21—F5	68.9 (3)
C3—C4—C5—N1	−31.9 (3)	C19—C18—C21—F6	128.6 (3)
C3—C2—C6—C7	−179.8 (2)	C17—C18—C21—F6	−49.4 (3)
C1—C2—C6—C7	−2.8 (4)	C27—C22—C23—C24	0.8 (4)
C2—C6—C7—C8	153.1 (3)	S1—C22—C23—C24	179.0 (2)
C2—C6—C7—C12	−28.6 (4)	C22—C23—C24—C25	−0.7 (4)
C12—C7—C8—C9	4.2 (4)	C23—C24—C25—C26	0.1 (4)

C6—C7—C8—C9	−177.4 (2)	C23—C24—C25—C28	179.8 (3)
C7—C8—C9—C10	−1.5 (4)	C24—C25—C26—C27	0.5 (4)
C8—C9—C10—C11	−1.9 (4)	C28—C25—C26—C27	−179.2 (3)
C8—C9—C10—C13	177.9 (2)	C25—C26—C27—C22	−0.4 (4)
C9—C10—C11—C12	2.5 (4)	C23—C22—C27—C26	−0.2 (4)
C13—C10—C11—C12	−177.3 (2)	S1—C22—C27—C26	−178.4 (2)
C10—C11—C12—C7	0.2 (4)	C2—C1—N1—C5	−66.0 (2)
C8—C7—C12—C11	−3.5 (4)	C2—C1—N1—S1	149.75 (17)
C6—C7—C12—C11	178.2 (2)	C4—C5—N1—C1	68.3 (2)
C11—C10—C13—F2	−53.8 (3)	C4—C5—N1—S1	−147.04 (17)
C9—C10—C13—F2	126.4 (3)	C1—N1—S1—O3	−171.68 (17)
C11—C10—C13—F3	−175.3 (2)	C5—N1—S1—O3	46.5 (2)
C9—C10—C13—F3	4.9 (4)	C1—N1—S1—O2	−42.1 (2)
C11—C10—C13—F1	64.7 (3)	C5—N1—S1—O2	176.04 (17)
C9—C10—C13—F1	−115.0 (3)	C1—N1—S1—C22	73.2 (2)
C3—C4—C14—C15	−174.5 (2)	C5—N1—S1—C22	−68.57 (19)
C5—C4—C14—C15	6.6 (4)	C23—C22—S1—O3	157.8 (2)
C4—C14—C15—C20	32.8 (4)	C27—C22—S1—O3	−24.0 (2)
C4—C14—C15—C16	−152.7 (3)	C23—C22—S1—O2	25.9 (2)
C20—C15—C16—C17	0.5 (4)	C27—C22—S1—O2	−155.88 (19)
C14—C15—C16—C17	−174.3 (2)	C23—C22—S1—N1	−88.2 (2)
C15—C16—C17—C18	1.6 (4)	C27—C22—S1—N1	90.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C29—H29*B*···O1	0.99	2.57	3.342 (4)	135
C29—H29*A*···O2^{i}	0.99	2.53	3.500 (4)	166
C27—H27···O3^{ii}	0.95	2.48	3.362 (3)	155
C5—H5*A*···Cl1^{iii}	0.99	2.81	3.776 (3)	166

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*, −*y*+2, −*z*+1; (iii) −*x*, −*y*+1, −*z*+1.