Electronic Supplementary material for:

Ground state structures of sulfate monoesters and sulfamates reveal similar reaction coordinates for sulfuryl and sulfamyl transfer

Emma Denehy, Jonathan M. White and Spencer J. Williams

School of Chemistry and Bio21 Biotechnology and Molecular Science Institute, University of Melbourne, Parkville, Australia 3010. Fax: +61 3 9347 5180; Tel: +61 3 8344 2422; E-mail: sjwill@unimelb.edu.au

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1. Synthesis of sulfate monoesters and sulfamate esters

Experimental

All melting points were obtained using an Electrothermal melting point apparatus and are uncorrected. IR spectra were obtained on BioRad FTS 165 FT-IR Spectrometer. ^{1}H and ^{13}C NMR spectra were obtained on a Varian UnityPlus400 instrument (399.8 MHz for ^{1}H, 100.5 MHz for ^{13}C). Internal standards employed for NMR spectra were (residual) chloroform (^{1}H, δ 7.24; ^{13}C, δ 77.0 ppm) or internal tetramethylsilane (^{1}H, ^{13}C, δ 0.00 ppm) for samples run in deuteriochloroform ($CDCl_3$); (residual) acetone (^{1}H, δ 2.05; ^{13}C, δ 29.9 ppm) for samples run in d_6-acetone; (residual) methanol (^{1}H, δ 4.87; ^{13}C, δ 49.15 ppm) for samples run in d_4-methanol and HOD (^{1}H, δ 4.63 ppm) for samples run in D_2O. Dioxane (^{13}C, δ 67.19 ppm) was used as an external standard for ^{13}C NMR spectra in D_2O. Flash chromatography was performed using Merck silica gel 60 (0.040-0.063 mm) according to the method described by Still *et al.*[1] Potassium methyl sulfate was obtained from Fluka.

General procedure for the synthesis of arylsulfate monoesters

The procedure described by Burkhardt and Lapworth,[2] with minor modifications, was employed in the synthesis of arylsulfamate monoesters. Briefly, sulfur trioxide pyridine complex (1 equiv.) was added to a stirred solution of the parent phenol (1.1 equiv.) in toluene (5-10 mL) under reflux. The solution was stirred under reflux overnight, during which time a thick, solid precipitate formed. Aqueous potassium hydroxide (0.1 M) was added until the precipitate dissolved and the aqueous phase was basic to universal indicator paper (0.1 M, 20-30 mL) and the mixture extracted with ethyl acetate (3 × 30 mL). The aqueous phase was acidified using glacial acetic acid and then extracted with ethyl acetate (3 × 30 mL). The aqueous phase was re-basified using aqueous potassium hydroxide (0.1 M), and the solvent evaporated under reduced pressure until a precipitate began to form. The precipitate was collected and a second crop obtained by further evaporation of the filtrate. The product was qualitatively tested for the presence of free sulfate by the dropwise addition of aqueous barium chloride (0.1 M) to an aqueous solution of the salt and was repeatedly recrystallised (0.1 M $KOH_{(aq)}$/ethanol unless specified otherwise) until no precipitate was evident.

Potassium 4-methoxyphenylsulfate monoester 1. 4-Methoxyphenol (2.09 g, 16.8 mmol) and sulfur trioxide pyridine complex (2.33 g, 14.7 mmol) according to the general procedure yielded potassium 4-methoxyphenylsulfate monoester as colourless iridescent plates (2.42 g, 68%), m.p. 234-241 °C with decomposition. ^{1}H NMR (400 MHz, D_2O) δ 3.65 (s, 3H, CH_3), 6.82 (app. d, 2H, Ar), 7.08 (app. d, 2H, Ar). ^{13}C NMR (100 MHz, D_2O) δ 58.7 (CH_3), 117.8,

125.9, 148.0, 159.9 (Ar). IR (KBr disc) ν 1509, 1280, 1267, 1257, 1241, 1065 (SO_2 sym), 841, 794 cm^{-1}.

Potassium 4-acetamidophenylsulfate monoester 3. 4-Acetamidophenol and sulfur trioxide pyridine complex according to the general procedure yielded potassium 4-acetamidophenylsulfate monoester as a finely divided, iridescent colourless solid, m.p. 247-252 °C (with evolution of gas). ^{1}H NMR (400 MHz, D_2O) δ 1.97 (s, 3H, CH_3), 7.12 (app. d, 2H, Ar), 7.24 (app. d, 2H, Ar). ^{13}C NMR (100 MHz, D_2O) δ 26.4 (CH_3), 125.7, 126.8, 138.4, 151.8 (Ar), 176.6 (C=O) IR (KBr disc) ν 3578 s (N-H), 1563, 1508, 1274, 1229, 1203, 1052 (SO_2 sym) cm^{-1}.

Potassium 4-nitrophenylsulfate monoester 4. 4-Nitrophenol (2.09 g, 16.8 mmol) and sulfur trioxide pyridine complex (2.08 g, 13.1 mmol) according to the general procedure yielded potassium 4-nitrophenylsulfate monoester as pale yellow needles (2.38 g, 70%), m.p. 248-251 °C. ^{1}H NMR (400 MHz, D_2O) δ 7.28 (app. d, 2H, Ar), 8.09 (app. d, 2H, Ar). ^{13}C NMR (100 MHz, D_2O) δ 125.4, 129.5, 148.7, 160.4 (Ar). IR (KBr disc) ν 1523, 1352, 1280, 1269, 1254, 1216, 1057 (SO_2 sym), 873, 726 cm^{-1}.

Potassium 2,2,2-trifluoroethylsulfate monoester 5. Potassium 2,2,2-trifluoroethylsulfate monoester was synthesized using a method adapted from Lloyd *et. al.*[3] Sulfur trioxide pyridine complex (7.89 g, 49.6 mmol) was added to a solution of 2,2,2-trifluoroethanol (3.64 mL, 5.00 g, 49.8 mmol) in toluene (10 mL) under reflux. The solution was stirred under reflux overnight (16 h), during which time a colourless precipitate formed. The reaction was allowed to cool to room temperature, and the supernatant was carefully decanted. The precipitate was dissolved in hot, freshly filtered, saturated barium hydroxide solution. Addition of further barium hydroxide solution to render the resulting solution slightly basic to universal indicator paper (pH 8-9) resulted in formation of a finely divided precipitate. The precipitate was removed by filtration through filter aid, and saturated potassium hydrogen carbonate (10 mL) was added to the filtrate. Upon standing, the excess barium carbonate precipitated rapidly as a finely divided solid, accompanied by vigorous evolution of gas. The precipitate was removed by filtration through a plug of filter aid. The solvent was removed from the filtrate under reduced pressure, yielding a colourless solid. The solid was extracted with 90% (v/v) aqueous ethanol (250 mL) at room temperature. The solvent was removed from the resultant solution by slow evaporation at room temperature to yield potassium 2,2,2-trifluoroethylsulfate monoester as large, colourless, transparent plates, m.p. 263-265 °C. ^{1}H

NMR (400 MHz, D_2O) δ 4.44 (q, 2H, CH_2, J = 8.4 Hz). ^{13}C NMR (100 MHz, D_2O) δ 68.3 (q, CH_2, J = 36 Hz), 127.0 (q, CF_3, J = 276 Hz). IR (KBr disc) ν 1264, 1231, 1174 (SO_2 asym), 1047 (SO_2 sym), 1038 cm^{-1}.

General procedure for the synthesis of arylsulfamates

Chlorosulfonyl isocyanate (CSI, 1 equiv.) was cautiously added to a stirred, heated solution of the phenol (1 equiv.) in toluene (5-10 mL) at reflux. The solution was stirred at reflux overnight, then cooled to 0 °C. Water was added dropwise to the stirred, cooled toluene solution until evolution of gas ceased, resulting in the formation of a precipitate. Unless otherwise described, the precipitate was collected by filtration, washed (toluene), dried, and recrystallised (toluene) to afford the desired sulfamate.

4-Methoxyphenylsulfamate ester 2. Addition of water to the solution of 4-methoxyphenol (3.00 g, 24.2 mmol) and CSI (3.42 g, 24.2 mmol) after overnight reflux resulted in precipitation of the product from toluene as an impure oil. The solvents were co-evaporated under reduced pressure and the resulting tan oil subjected to flash chromatography (20% ethyl acetate: 80 % petroleum spirit) to yield a pale oil. The oil was repeatedly extracted with hot toluene until the impurity dispersed as a flocculent black solid. The solid was removed by filtration, and the filtrate combined with the washings and concentrated to the point of precipitation by heating to afford 4-methoxyphenylsulfamate ester as colourless finely divided iridescent needles (568 mg, 18%), m.p. 61-63 °C (lit.[4] 62-64 °C). ^1H NMR (400 MHz, $CDCl_3$) δ 3.80 (s, 3H, CH_3), 5.10 (br s, 2H, NH_2), 6.89 (app. d, 2H, Ar), 7.25 (app. d, 2H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$) δ 56.7 (Me), 115.9, 159.5, 124.3, 144.5 (Ar). IR (KBr disc) ν 3376 (N-H), 3276 (N-H), 1503, 1363 (SO_2 asym), 1245, 1165 (SO_2 sym), 1152, 836 cm^{-1}.

4-Nitrophenylsulfamate ester 7. 4-Nitrophenol (2.00 g, 14.3 mmol) and CSI (2.00 g, 14.3 mmol) according to the general procedure yielded 4-nitrophenylsulfamate ester as fine beige needles (1.6 g, 52%), m.p. 104-105.5 °C (lit.[5] 100-102 °C). ^1H NMR (400 MHz, d_6-acetone) δ 7.46 (s, 2H, NH_2), 7.57 (app. d, 2H, Ar), 8.32 (app. d, 2H, Ar). ^{13}C NMR (100 MHz, d_6-acetone) δ 123.0, 125.4, 145.9, 155.1 (Ar). IR (KBr disc) ν 3424 (N-H), 3296 (N-H), 1519, 1405, 1354 (SO_2 asym), 1177 (SO_2 sym) cm^{-1}.

3-Nitrophenylsulfamate ester 8. 3-Nitrophenol (2.00 g, 14.3 mmol) and CSI (2.03 g, 14.3 mmol) according to the general procedure yielded 3-nitrophenylsulfamate ester as pale brown needles (1.5 g, 48%), m.p. 119-120 °C (lit.[4] 118-120 °C). ^1H NMR (400 MHz, d_6-acetone) δ

7.42 (br s, 2H, NH_2), 7.75-7.78 (m, 2H, Ar), 8.16 (s, 1H, Ar), 8.21 (dd, 1H, Ar). ^{13}C NMR (100 MHz, d_6-acetone) δ 117.8, 121.8, 129.3, 131.3, 149.1, 151.1 (Ar). IR (KBr disc) ν 3413 (N-H), 3312 (N-H), 1531, 1391, 1378 (SO_2 asym), 1182 (SO_2 sym), 834 cm^{-1}.

4-Iodophenylsulfamate ester 9. 4-Iodophenol (1.50 g, 6.82 mmol) and CSI (0.96 g, 6.78 mmol) according to the general procedure yielded 4-iodophenylsulfamate ester as colourless needles (1.35 g, 67%), m.p. 142-144 °C (lit.[5] 134-136 °C) IR (KBr disc) ν 3378 (N-H), 3275 (N-H), 1478, 1352 s (SO_2 asym), 1176 (SO_2 sym), 1158, 876 cm^{-1}.

4-Cyanophenylsulfamate ester 10. 4-Cyanophenol (2.0 g; 16.9 mmol) and CSI (1.46 mL; 16.9 mmol) according to the general procedure yielded 4-cyanophenylsulfamate ester as beige needles, m.p. 152-153 °C (lit.[5] mp 152-153 °C). ^1H NMR (400 MHz, d_4-methanol) δ 7.46 (app. d, 2H, Ar), 7.80 (app. d, 2H, Ar). ^{13}C NMR (100 MHz, d_4-methanol) δ 111.5 (Ar), 119.2 (C≡N), 124.4, 135.3, 155.6 (Ar). IR (KBr disc) ν 3358 (N-H), 3256 (N-H), 2241, (C≡N) 1382 s (SO_2 asym), 1187 s (SO_2 sym), 1160, 870 cm^{-1}.

4-Chlorophenylsulfamate ester 11. 4-Chlorophenol (2.00 g, 15.6 mmol) and CSI (2.20 g, 15.6 mmol) according to the general procedure yielded 4-chlorophenylsulfamate ester as cream, finely divided iridescent needles (1.62 g, 49%), m.p. 103-104 °C (lit.[4] 103-105 °C). ^1H NMR (400 MHz, $CDCl_3$) δ 5.17 (s, 2H, NH_2), 7.25 (app. d, 2H, Ar), 7.36 (app. d, 2H, Ar). ^{13}C NMR (100 MHz, d_6-acetone) δ 123.5, 130.0, 133.0, 148.3 (Ar). IR (KBr disc) ν 3385 (N-H), 3277 (N-H), 1486, 1366 (SO_2 asym), 1180, 1170, 1161 (SO_2 sym), 869, 841 cm^{-1}.

3-Chlorophenylsulfamate ester 12. 3-Chlorophenol (1.2 g, 9.3 mmol) and CSI (1.35 g, 9.3 mmol) according to the general procedure yielded 3-chlorophenylsulfamate ester as colourless needles (258 mg, 13%), m.p. 82-83 °C (lit.[4] 81-82 °C). ^1H NMR δ 5.12 (br s, 2H, NH_2), 7.23-7.38 (m, 4H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$) δ 120.4, 122.7, 127.7, 130.6, 135.1, 150.3 (Ar). IR (KBr disc) ν 3377 (N-H), 3277 (N-H), 1586, 1466, 1378 (SO_2 asym), 1170 (SO_2 sym), 911 cm^{-1}.

Phenylsulfamate ester 13. Phenol (2.00 g, 21.2 mmol) and CSI (3.07 g, 21.2 mmol) according to the general procedure yielded phenylsulfamate ester as colourless iridescent plates (759 mg, 21%), m.p. 83-84 °C (lit.[4] 81-85 °C). ^1H NMR δ (400 MHz, $CDCl_3$) δ 5.01 (br s, 2H, NH_2), 7.31-7.44 (m, 5H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$) δ 122.1, 127.4, 129.9,

150.0 (Ar). IR (KBr disc) ν 3422 (N-H), 3307 (N-H), 1531, 1391, 1378 (SO_2 asym), 1182 (SO_2 sym), 834 cm^{-1}.

3,4-Dinitrophenylsulfamate ester 14. Chlorosulfonyl isocyanate (0.8 mL, 1.30 g, 9.19 mmol) was cautiously added to a stirred, heated solution of the parent phenol (520 mg, 2.82 mmol) in toluene (10 mL) at reflux. The solution was stirred at reflux overnight, then cooled to room temperature. The solvent was removed under reduced pressure and the creamy residue cooled to 0 °C. Cold aqueous acetonitrile (149 μL H_2O in 5 mL acetonitrile) was added to the cooled residue. The solvents were removed under reduced pressure and the solid recrystallised from chloroform to yield 3,4-dinitrophenylsulfamate ester as dark yellow cubic crystals, (445 mg, 60%), m.p. 92-95 °C. ^{1}H NMR (400 MHz, d_6-acetone) δ 7.91 (dd, 1H, H6, *J* = 9.0, 2.4 Hz), 8.10 (d, 1H, H2, *J* = 2.4 Hz), 8.33 (d, 1H, H5, *J* = 8.8 Hz). ^{13}C NMR (100 MHz, d_6-acetone) δ 120.1, 127.9, 128.3, 141.3, 145.2, 155.1 (Ar). IR (KBr disc) ν 3429 (N-H), 3321 (N-H), 1549, 1396 (SO_2 asym), 1372, 1186 (SO_2 sym), 833 cm^{-1}.

Preparation of sulfamoyl chloride. The general method of Appel and Berger,[6] with slight modification similar to that described by Geisler *et al*.[7] was used in the preparation of sulfamoyl chloride. Formic acid (1 equiv.) was added to a chilled solution of chlorosulfonyl isocyanate (1 equiv.) in acetonitrile. The mixture was stirred at room temperature under an atmosphere of nitrogen (2 h) before the addition of catalytic *N,N*-dimethylacetamide (0.05 mL). The mixture was stirred for a further hour then was used immediately in the next step.

General procedure for the synthesis of alkylsulfamate esters

Sulfamoylation was performed following the method of Okada *et al*.[8] A solution of the appropriate alcohol in *N,N*-dimethylacetamide was added to the chilled, freshly prepared sulfamoyl chloride solution. The solution was stirred overnight at room temperature under an atmosphere of nitrogen, after which the reaction was quenched with water. The product was extracted into ethyl acetate (3 × 50 mL), and organic extracts combined and washed with water (3 × 100 mL), dried ($MgSO_4$), and the solvent removed under reduced pressure. Vacuum sublimation of the crude material yielded crystals of suitable quality for X-ray analysis.

Ethylsulfamate ester 15. Vacuum sublimation at 35 °C of the crude material from the reaction of sulfamoyl chloride and ethanol according to the general procedure yielded the ethylsulfamate ester as colourless, finely divided cubic crystals, m.p. 40.5-42 °C (lit.[9] m.p. 37-

39 °C). ^{1}H NMR (400 MHz, d$_4$-methanol) δ 1.37 (t, 3H, CH_3, J = 7.2 Hz), 4.19 (q, 2H, CH_2, J = 7.2 Hz). ^{13}C NMR (100 MHz, d$_4$-methanol) δ 15.1 (CH_3), 67.3 (CH_2). IR (KBr disc) ν 3371 (N-H), 3289 (N-H), 1353 (SO_2 asym), 1181 (SO_2 sym), 1010, 923 cm^{-1}.

2,2,2-Trifluoroethylsulfamate ester 16. Vacuum sublimation at 45-50 °C of the crude material from the reaction of sulfamoyl chloride and 2,2,2-trifluoroethanol according to the general procedure yielded the 2,2,2-trifluoroethylsulfamate ester as colourless, finely divided cubic crystals, m.p. 60.5-62 °C. ^{1}H NMR (400 MHz, d$_4$-methanol) δ 4.55 (q, 2H, CH_2, J = 8 Hz). ^{13}C NMR (100 MHz, d$_4$-methanol) δ 63.7 (q, CH_3,, J = 37 Hz), 122.2 (q, CF_3, J = 275 Hz). IR (KBr disc) ν 3392 (N-H), 3288 (N-H), 1370 (SO_2 asym), 1309, 1276, 1184 (SO_2 sym), 1054, 966 cm^{-1}.

2. Explanation of calculations

The length of the observed bond as a function of leaving group pK_a value is given by equation (1) and the dependence of reaction rate on leaving group pK_a value (the Brønsted relationship) by equation (2):

$$X - O_b = \alpha(pK_a) + c_1 \qquad (1)$$

$$\log k = \beta(pK_a) + c_2 \qquad (2)$$

where α is the gradient of the structure/reactivity plot (expressed in units of Å) and β is the gradient of the LFER plot.

The rate term in (2) can be expressed as an energy term using equation (3). Substitution of (2) into (3) gives equation (4), the energetic dependence of leaving group ability:

$$\Delta G = -2.3RT \log k \qquad (3)$$

$$\Delta G = -2.3RT\beta(pK_a) + c_3 \qquad (4)$$

(4) has the units of cal mol^{-1} or J mol^{-1}, depending upon the value used for R.

Rearranging (1) in terms of pK_a yields (5), which, upon substitution into (4), yields (6), a relationship that describes the energetic cost of stretching a bond as a function of bond length:

$$pK_a = \frac{(X - O_b) - c_1}{\alpha} \qquad (5)$$

$$\Delta G = -\frac{2.3RT\beta}{\alpha}(X - O_b) + c_4 \qquad (6)$$

Note: the experimental values for phosphate monoester hydrolysis used are α = -8 × 10^{-3} Å, β = -1.23 and T = 312 K;[10] those for sulfate monoester hydrolysis are α = -4.2 × 10^{-3} Å, β = -1.2 and T = 373 K.[11]

3. Crystallographic materials

The temperature was maintained at 130.0(2) using an Oxford Cryostream cooling device. Intensity data were collected with a Bruker SMART Apex CCD detector using Mo KR radiation (graphite crystal monochromator λ = 0.71073). Data were reduced using the program SAINT.[12] The structures were solved by direct methods and difference Fourier synthesis.

Crystallisation conditions for growth of single crystals.

Sulfate monesters:

4-nitrophenyl (**4**)	0.1 M KOH (aq)/EtOH, vapour diffusion, 4 ºC	pK_a = 7.15
4-acetamidophenyl (**3**)	0.1 M KOH (aq)/EtOH, vapour diffusion, 4 ºC	pK_a = 9.58
methyl (**6**)	H_2O/acetone, vapour diffusion, 4 ºC	pK_a = 15.6
4-methoxyphenyl (**1**)	Aqueous methanol, room temp.	pK_a = 10.21
2,2,2-trifluoroethyl (**5**)	90% EtOH (aq), slow evaporation at room temp.	pK_a = 12.37

Sulfamate esters:

4-nitrophenyl (**7**)	EtOAc/pet. Spirit, vapour diffusion, room temp.	pK_a = 7.15
4-methoxyphenyl (**2**)	EtOAc/pet. Spirit, vapour diffusion, room temp.	pK_a = 10.21
3-nitrophenyl (**8**)	Toluene, slow cooling	pK_a = 8.36
3-chlorophenyl (**12**)	Toluene, slow cooling	pK_a = 9.12
4-chlorophenyl (**11**)	EtOAc/pet. Spirit, vapour diffusion, room temp.	pK_a = 9.41
4-cyanophenyl (**10**)	Ethyl acetate, slow evaporation at room temp.	pK_a = 7.79
phenyl (**13**)	EtOAc/pet. Spirit, vapour diffusion, room temp.	pK_a = 9.99
4-iodophenyl (**9**)	Toluene, slow diffusion	pK_a = 9.45
2,2,2-trifluoroethyl (**16**)	Vacuum sublimation	pK_a = 12.37
ethyl (**15**)	Vacuum sublimation	pK_a = 16
3,4-dinitrophenyl (**14**)	Chloroform, slow cooling	pK_a = 5.36

Summary table of crystallographic data

Compound number		**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Chemical formula		$C_7H_7KO_5S$	$C_7H_9NO_4S$	$C_8H_{10}KNO_6S$	$C_6H_4KO_6S$	$C_2H_2F_3KO_4S$	CH_3KO_4S	$C_6H_6N_2O_5S$	$C_6H_6N_2O_5S$
Formula weight		242.29	203.21	287.33	257.26	218.20	150.19	218.19	218.19
Crystal system		Monoclinic	Orthorhombic	Triclinic	Orthorhombic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Unit cell dimensions (Å)	a	7.1098(4)	4.9816(4)	6.7995(5)	6.9153(4)	11.862(3)	9.1599(13)	9.9687(10)	10.8417(13)
	b	38.923(2)	7.0344(5)	6.9857(5)	13.1212(7)	7.0333(16)	6.9471(10)	6.9592(7)	8.2431(10)
	c	7.1463(4)	25.1874(18)	12.1695(9)	18.9869(10)	9.150(2)	7.3311(10)	12.2538(12)	10.2599(13)
α °		90	90	89.0620(10)	90	90	90	90	90
β °		93.3540(10)	90	81.3830(10)	90	107.962(4)	93.147(2)	98.383(2)	116.410(2)
γ °		90	90	79.6480(10)	90	90	90	90	90
Unit cell volume ($Å^3$)		1974.2(2)	882.63(11)	562.18(7)	1722.82(16)	726.1(3)	465.81(11)	841.01(15)	821.22(17)
Temperature (K)		130(2)	130(2)	130(2)	130(2)	130(2)	130(2)	130(2)	130(2)
Space group		$P2_1/c$	$P2_12_12_1$	P-1	Pbca	$P2_1/c$	$P2_1/c$	$P2_1/n$	$P2_1/c$
No. formula units in unit cell (Z)		8	4	2	8	4	4	4	4
Linear absorption coefficient μ (mm^{-1})		0.741	0.348	0.674	0.867	1.042	1.482	0.384	0.393
Number of independent reflections (R_{int})		4444 (0.0276)	2110 (0.0478)	1957 (0.0613)	1514 (0.0999)	1271 (0.0252)	1045 (0.0217)	1963 (0.0848)	1862 (0.0157)
Final *R* indices [I>2σ(I)]		R1 = 0.0329 wR2 = 0.0784	R1 = 0.0286 wR2 = 0.0733	R1 = 0.0319 wR2 = 0.0818	R1 = 0.0264 wR2 = 0.0734	R1 = 0.0451 wR2 = 0.1220	R1 = 0.0292 wR2 = 0.0811	R1 = 0.0348 wR2 = 0.0972	R1 = 0.0315 wR2 = 0.0896
R indices (all data)		R1 = 0.0371 wR2 = 0.0806	R1 = 0.0296 wR2 = 0.0739	R1 = 0.0326 wR2 = 0.0824	R1 = 0.0275 wR2 = 0.0743	R1 = 0.0473 wR2 = 0.1237	R1 = 0.0299 wR2 = 0.0818	R1 = 0.0363 wR2 = 0.0986	R1 = 0.0333 wR2 = 0.0911

Compound number		**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**
Chemical formula		$C_6H_6INO_3S$	$C_7H_6N_2O_3S$	$C_6H_6ClNO_3S$	$C_6H_6ClNO_3S$	$C_6H_7NO_3S$	$C_6H_5N_3O_7S$	$C_2H_7NO_3S$	$C_2H_4F_3NO_3S$
Formula weight		299.08	198.20	207.63	207.63	173.19	263.19	125.15	179.12
Crystal system		Orthorhombic	Monoclinic	Monoclinic	Monoclinic	Orthorhombic	Monoclinic	Orthorhombic	Triclinic
Unit cell dimensions (Å)	a	22.457(6)	4.9239(5)	4.8116(3)	5.0414(9)	8.0387(6)	14.7131(8)	8.9053(11)	4.8598(12)
	b	8.135(2)	14.8822(14)	20.2367(14)	6.9625(13)	5.3810(4)	6.9420(4)	8.2409(11)	4.9203(12)
	c	4.7648(12)	11.2090(11)	8.5704(6)	11.813(2)	16.5114(12)	9.7229(6)	14.2486(18)	13.834(4)
α °		90	90	90	90	90	90	90	83.262(4)
β °		90	96.432(2)	94.7730(10)	92.276(4)	90	103.8040(10)	90	87.319(4)
γ °		90	90	90	90	90	90	90	64.377(4)
Unit cell volume ($Å^3$)		870.5(4)	816.21(14)	831.61(10)	414.30(13)	714.22(9)	964.40(10)	1045.7(2)	296.21(13)
Temperature		130(2)	130(2)	130(2)	130(2)	130(2)	130(2)	130(2)	130(2)
Space group		$Pna2_1$	$P2_1/c$	$P2_1/n$	P2(1)	$Pca2_1$	$P2_1/c$	Pbca	P-1
No. formula units in unit cell (Z)		4	4	4	2	4	4	8	2
Linear absorption coefficient μ (mm^{-1})		3.884	0.369	0.673	0.676	0.405	0.396	0.518	0.560
Number of independent reflections (R_{int})		1751 (0.0290)	1863 (0.0342)	1876 (0.0145)	1796 (0.0190)	1611 (0.0533)	1694 (0.0286)	1202 (0.0519)	1020 (0.0989)
Final *R* indices [I>2σ(I)]		R1 = 0.0286 wR2 = 0.0727	R1 = 0.0375 wR2 = 0.1008	R1 = 0.0286 wR2 = 0.0883	R1 = 0.0258 wR2 = 0.0673	R1 = 0.0312 wR2 = 0.0824	R1 = 0.0296 wR2 = 0.0815	R1 = 0.0304 wR2 = 0.0852	R1 = 0.0487 wR2 = 0.1269
R indices (all data)		R1 = 0.0289 wR2 = 0.0730	R1 = 0.0415 wR2 = 0.1035	R1 = 0.0298 wR2 = 0.0894	R1 = 0.0265 wR2 = 0.0677	R1 = 0.0315 wR2 = 0.0826	R1 = 0.0301 wR2 = 0.0819	R1 = 0.0323 wR2 = 0.0868	R1 = 0.0524 wR2 = 0.1288

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