Supporting information

Synthesis and aromatisation of cyclic enediyne-containing amino acids

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Contents

Crystal data and structure refinement for COMPOUND 4.

Crystal colour	colorless transparant
Crystal shape	regular fragment
Crystal size	0.31 x 0.15 x 0.15 mm
Empirical formula	C22 H19 N O4 S
Formula weight	393.44
Temperature	208(2) K
Radiation / Wavelength	MoK\a (graphite mon.) / 0.71073 A
Crystal system, space group	Triclinic, P -1
Unit cell dimensions 23786 reflections 3.020 < theta < 27.500)	a, alp= 6.8047(2) A, 111.921(6) deg. b, bet= 11.9451(7) A, 94.992(5) deg. c, gam= 12.7864(9) A, 92.605(4) deg.
Volume	957.09(9) A^3
Z, Calculated density	2, 1.365 Mg/m^3
Absorption coefficient	0.198 mm^-1
Diffractometer / scan	Enraf-Nonius CAD4 / area detector \f and \w scan
F(000)	412
Theta range for data collection	3.02 to 27.50 deg.
Index ranges	-8<=h<=8, -15<=k<=15, -16<=l<=16
Reflections collected / unique	23786 / 4377 [R(int) = 0.0249]
Reflections observed	3612 ([Io>2sigma(Io)])
Absorption correction	Semi-empirical from psi-scans
Range of relat. transm. factors	1.000 and 1.000
Refinement method	Full-matrix least-squares on F^2
Computing	SHELXL-97 (Sheldrick, 1997)
Data / restraints / parameters	4377 / 0 / 329
Goodness-of-fit on F^2	1.038
SHELXL-97 weight parameters	weight pars
Final R indices [I>2sigma(I)]	R1 = 0.0373, wR2 = 0.0935
R indices (all data)	R1 = 0.0494, wR2 = 0.0995
Largest diff. peak and hole	0.367 and -0.341 e.A^-3

Crystal data and structure refinement for COMPOUND 19.

Crystal colour	translucent light yellow
Crystal shape	rather regular large fragment
Crystal size	0.63 x 0.46 x 0.33 mm
Empirical formula	C24 H23 N O4 S
Formula weight	421.49
Temperature	208(2) K
Radiation / Wavelength	MoKa (graphite mon.) / 0.71073 A
Crystal system, space group	Triclinic, P -1
Unit cell dimensions 115 reflections 2.580 < theta < 27.500)	a, alp= 8.4021(5) A, 69.128(6) deg. b, bet= 11.9570(9) A, 70.351(5) deg. c, gam= 12.0405(8) A, 84.942(5) deg.
Volume	1063.76(12) A^3
Z, Calculated density	2, 1.316 Mg/m^3
Absorption coefficient	0.183 mm^-1
Diffractometer / scan	Nonius KappaCCD with area detector f and w scan
F(000)	444
Theta range for data collection	2.58 to 27.50 deg.
Index ranges	-10<=h<=10, -15<=k<=15, -15<=l<=15
Reflections collected / unique	27752 / 4862 [R(int) = 0.0199]
Reflections observed	4028 ([Io>2sigma(Io)])
Completeness to 2theta = 25.00	99.6%
Absorption correction	SADABS multiscan correction (Sheldrick, 1996)
Refinement method	Full-matrix least-squares on F^2
Computing	SHELXL-97 (Sheldrick, 1997)
Data / restraints / parameters	4862 / 0 / 273
Goodness-of-fit on F^2	1.083
SHELXL-97 weight parameters	0.0407, 0.4145
Final R indices [I>2sigma(I)]	R1 = 0.0364, wR2 = 0.0888
R indices (all data)	R1 = 0.0486, wR2 = 0.0967
Largest diff. peak and hole	0.238 and -0.390 e.A^-3

Crystal data and structure refinement for COMPOUND 29.

Crystal colour	translucent colourless
Crystal shape	regular needle
Crystal size	0.22 x 0.05 x 0.03 mm
Empirical formula	C20 H21 N O4 S
Formula weight	371.44
Temperature	208(2) K
Radiation / Wavelength	MoKa (graphite mon.) / 0.71073 A
Crystal system, space group	Orthorhombic, P b c a
Unit cell dimensions 110 reflections 2.290 < theta < 22.000)	a, alp= 8.6566(7) A, 90 deg. b, bet= 12.0779(8) A, 90 deg. c, gam= 35.500(3) A, 90 deg.
Volume	3711.7(5) A^3
Z, Calculated density	8, 1.329 Mg/m^3
Absorption coefficient	0.199 mm^-1
Diffractometer / scan	Nonius KappaCCD with area detector f and w scan
F(000)	1568
Theta range for data collection	2.29 to 22.00 deg.
Index ranges	-9<=h<=9, -12<=k<=12, -37<=l<=37
Reflections collected / unique	30562 / 2279 [R(int) = 0.1411]
Reflections observed	1648 ([Io>2sigma(Io)])
Completeness to 2theta = 22.00	97.9%
Absorption correction	SADABS multiscan correction (Sheldrick, 1996)
Refinement method	Full-matrix least-squares on F^2
Computing	SHELXL-97 (Sheldrick, 1997)
Data / restraints / parameters	2279 / 0 / 250
Goodness-of-fit on F^2	1.224
SHELXL-97 weight parameters	0.0404, 5.6577
Final R indices [I>2sigma(I)]	R1 = 0.0763, wR2 = 0.1329
R indices (all data)	R1 = 0.1146, wR2 = 0.1452
Largest diff. peak and hole	0.225 and -0.246 e.A^-3

Crystal data and structure refinement for COMPOUND 34.

Crystal colour	translucent colourless
Crystal shape	rather regular fragment
Crystal size	0.18 x 0.11 x 0.04 mm
Empirical formula	C23 H21 N O4 S
Formula weight	407.47
Temperature	208(2) K
Radiation / Wavelength	MoKa (graphite mon.) / 0.71073 A
Crystal system, space group	Monoclinic, P 21
Unit cell dimensions 43 reflections 2.570 < theta < 25.000)	a, alp= 8.1942(12) A, 90 deg. b, bet= 9.7320(7) A, 104.996(9) deg. c, gam= 13.0079(13) A, 90 deg.
Volume	1002.00(19) A^3
Z, Calculated density	2, 1.351 Mg/m^3
Absorption coefficient	0.191 mm^-1
Diffractometer / scan	Nonius KappaCCD with area detector f and w scan
F(000)	428
Theta range for data collection	2.57 to 25.00 deg.
Index ranges	-9<=h<=9, -11<=k<=11, -15<=l<=15
Reflections collected / unique	13482 / 3516 [R(int) = 0.0488]
Reflections observed	2608 ([Io>2sigma(Io)])
Completeness to 2theta = 25.00	99.8%
Absorption correction	SADABS multiscan correction (Sheldrick, 1996)
Refinement method	Full-matrix least-squares on F^2
Computing	SHELXL-97 (Sheldrick, 1997)
Data / restraints / parameters	3516 / 1 / 264
Goodness-of-fit on F^2	1.080
SHELXL-97 weight parameters	0.0202, 0.4542
Final R indices [I>2sigma(I)]	R1 = 0.0523, wR2 = 0.0752
R indices (all data)	R1 = 0.0867, wR2 = 0.0833
Absolute structure parameter	0.07(10)
Largest diff. peak and hole	0.224 and -0.264 e.A^-3