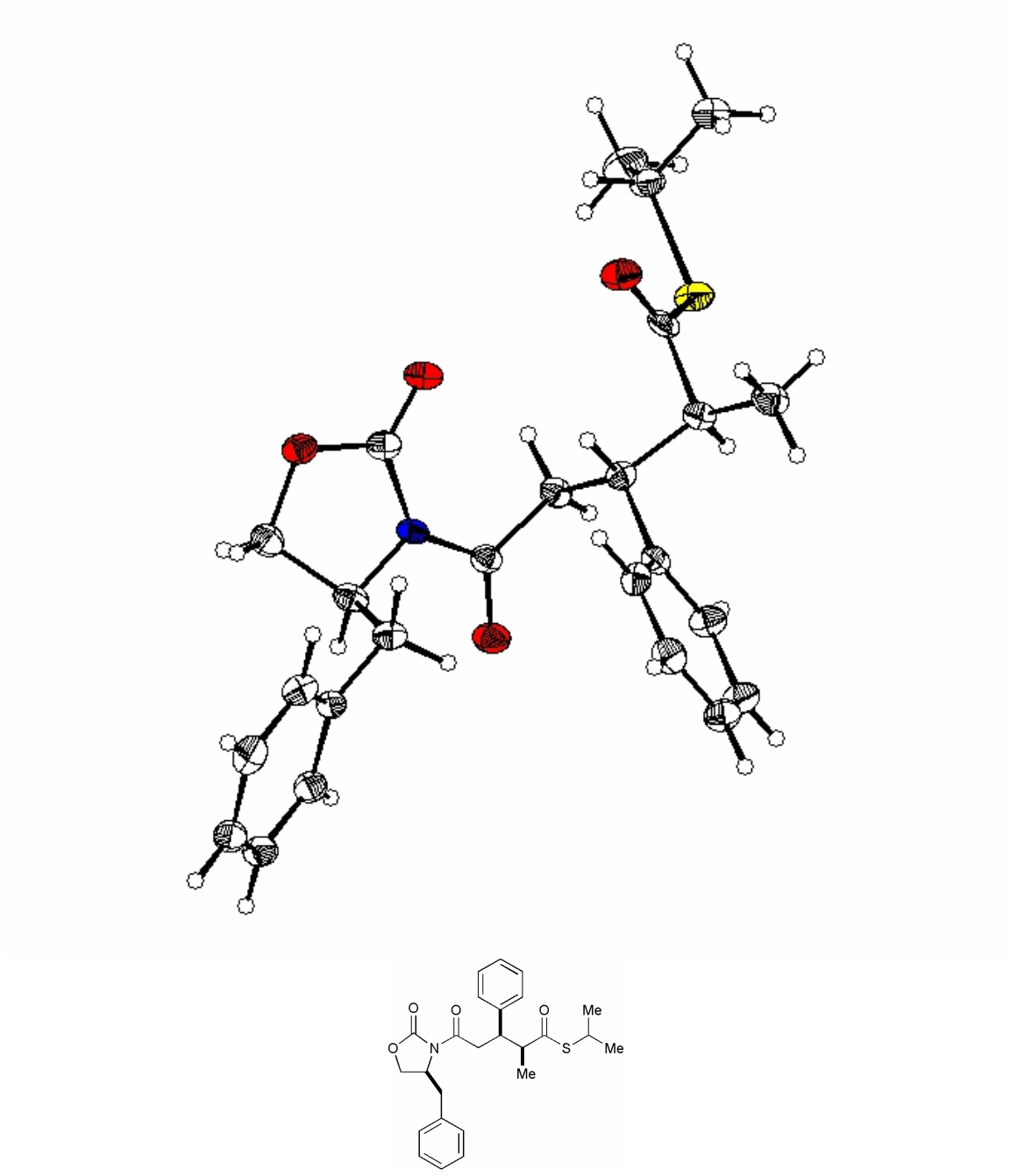
Appendix 1.

X-Ray Crystallographic Data for (2*S*, 3*R*)-5-((4*S*)-4-Benzyl-2-oxo-oxazolidin-3-yl)-2-methyl-5-oxo-3-phenyl-pentanethioic acid *S*-isopropyl ester



CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 07 July 2004

Crystal Structure Analysis of:

(2*S*, 3*R*)-5-((4*S*)-4-Benzyl-2-oxo-oxazolidin-3-yl)-2-methyl-5-oxo-3-phenyl-pentanethioic acid *S*-isopropyl ester (CJB12)

(shown below)

For Investigator: Chris Borths

Advisor: D. W. C. MacMillan

By Michael W. Day

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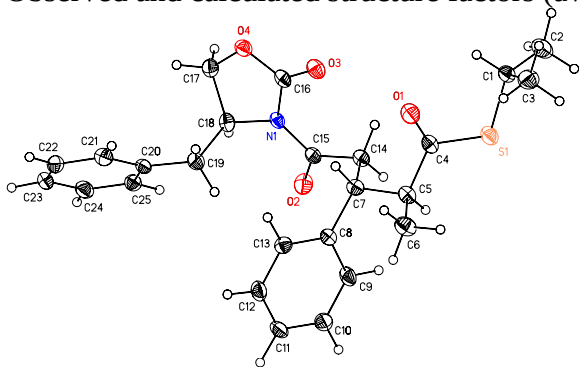


CJB12

Note: Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 239502.

Table 1. Crystal data and structure refinement for CJB12 (CCDC 239502).

Empirical formula	$C_{25}H_{29}NO_4S$
Formula weight	439.55
Crystallization Solvent	THF/hexanes
Crystal Habit	Column
Crystal size	0.48 x 0.17 x 0.13 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 5769 reflections used in lattice determination	2.86 to 27.99°
Unit cell dimensions	a = 12.701(3) Å b = 6.4520(13) Å β= 107.706(3)° c = 14.854(3) Å
Volume	1159.5(4) $Å^3$
Z	2
Crystal system	Monoclinic
Space group	$P2_1$
Density (calculated)	1.259 Mg/m^3
F(000)	468
Data collection program	Bruker SMART v5.054
θ range for data collection	1.68 to 27.99°
Completeness to θ = 27.99°	93.5 %
Index ranges	$-16 \le h \le 16, -8 \le k \le 8, -19 \le l \le 19$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.45
Reflections collected	13847
Independent reflections	5068 [R_{int}= 0.0709]
Absorption coefficient	0.170 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9782 and 0.9227

Table 1 (cont.)

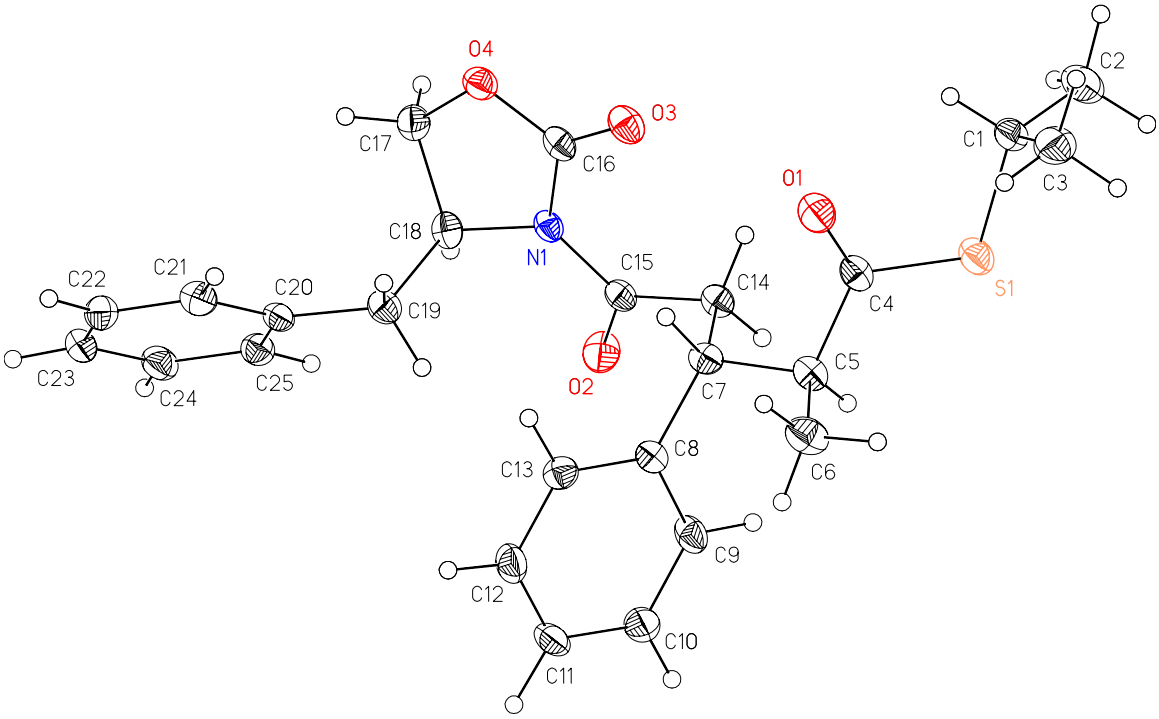
Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	5068 / 1 / 396
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.097
Final R indices [$I>2\sigma(I)$, 3973 reflections]	R1 = 0.0415, *w*R2 = 0.0648
R indices (all data)	R1 = 0.0569, *w*R2 = 0.0677
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Absolute structure parameter	0.03(6)
Largest diff. peak and hole	0.347 and -0.260 e.Å^{-3}

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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O3
C17
C16
C18
N1
C15
C14
O1
C4
S1
C2
C1
C3
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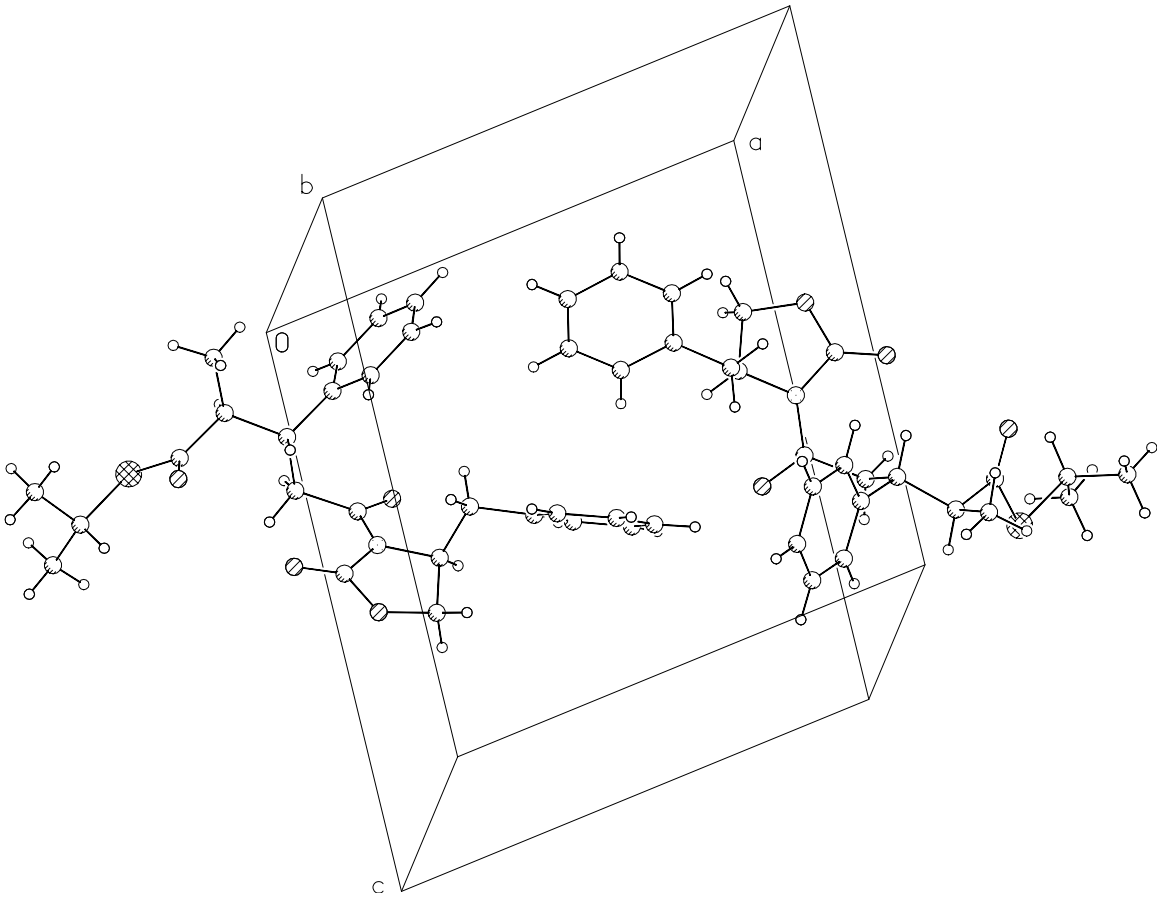
C9
C12
C10
C11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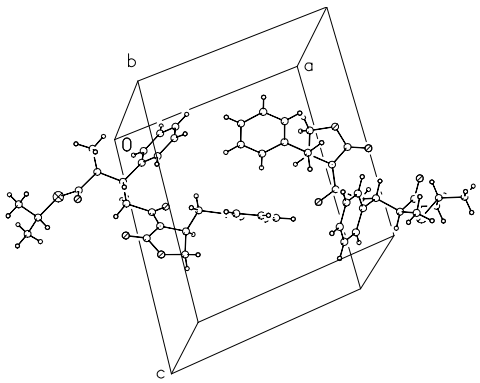
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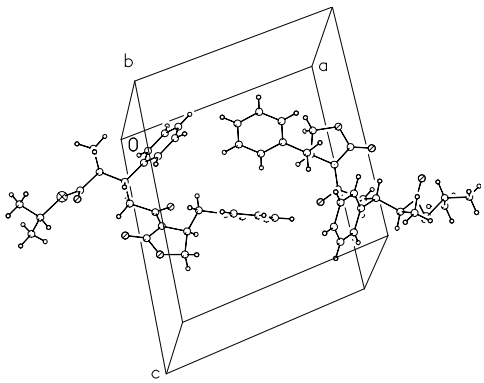
b
a
O
c

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for CJB12 (CCDC 239502). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
S(1)	-3118(1)	5719(1)	6145(1)	26(1)
O(1)	-2767(1)	9144(2)	7180(1)	28(1)
O(2)	1801(1)	6260(2)	8408(1)	28(1)
O(3)	-837(1)	8606(2)	9285(1)	25(1)
O(4)	553(1)	8885(2)	10641(1)	22(1)
N(1)	1006(1)	7921(3)	9364(1)	17(1)
C(1)	-4286(2)	5646(4)	6632(2)	24(1)
C(2)	-4612(2)	3383(4)	6699(2)	35(1)
C(3)	-5239(2)	6959(5)	6040(2)	33(1)
C(4)	-2452(2)	8061(3)	6650(2)	20(1)
C(5)	-1460(2)	8592(4)	6318(2)	21(1)
C(6)	-1754(2)	10534(4)	5691(2)	30(1)
C(7)	-414(2)	8901(3)	7183(2)	19(1)
C(8)	548(2)	9739(3)	6884(1)	18(1)
C(9)	969(2)	8683(4)	6248(2)	24(1)
C(10)	1829(2)	9494(4)	5962(2)	28(1)
C(11)	2280(2)	11414(4)	6305(2)	26(1)
C(12)	1884(2)	12480(4)	6944(2)	26(1)
C(13)	1027(2)	11643(3)	7231(2)	22(1)
C(14)	-144(2)	6838(3)	7733(2)	19(1)
C(15)	948(2)	6944(3)	8509(1)	19(1)
C(16)	139(2)	8475(3)	9708(2)	19(1)
C(17)	1745(2)	8513(4)	10971(2)	23(1)
C(18)	2083(2)	8361(3)	10067(1)	20(1)
C(19)	2566(2)	10381(4)	9804(2)	21(1)
C(20)	3669(2)	11005(3)	10481(1)	19(1)
C(21)	3796(2)	12904(3)	10956(2)	23(1)
C(22)	4822(2)	13528(4)	11540(2)	27(1)
C(23)	5746(2)	12277(4)	11669(2)	27(1)
C(24)	5629(2)	10374(4)	11214(2)	25(1)
C(25)	4603(2)	9743(4)	10621(2)	21(1)

Table 3. Bond lengths [Å] and angles [°] for CJB12 (CCDC 239502).

S(1)-C(4)	1.782(2)	C(20)-C(21)	1.398(3)
S(1)-C(1)	1.838(2)	C(20)-C(25)	1.402(3)
O(1)-C(4)	1.208(2)	C(21)-C(22)	1.388(3)
O(2)-C(15)	1.220(2)	C(21)-H(21)	0.95(2)
O(3)-C(16)	1.210(2)	C(22)-C(23)	1.388(3)
O(4)-C(16)	1.351(2)	C(22)-H(22)	0.93(2)
O(4)-C(17)	1.462(2)	C(23)-C(24)	1.388(3)
N(1)-C(16)	1.394(2)	C(23)-H(23)	0.97(2)
N(1)-C(15)	1.400(3)	C(24)-C(25)	1.393(3)
N(1)-C(18)	1.474(3)	C(24)-H(24)	0.95(2)
C(1)-C(3)	1.518(3)	C(25)-H(25)	0.93(3)
C(1)-C(2)	1.529(4)		
C(1)-H(1)	0.97(2)	C(4)-S(1)-C(1)	101.09(11)
C(2)-H(2A)	1.02(3)	C(16)-O(4)-C(17)	110.60(15)
C(2)-H(2B)	1.01(3)	C(16)-N(1)-C(15)	128.21(16)
C(2)-H(2C)	1.02(3)	C(16)-N(1)-C(18)	110.95(17)
C(3)-H(3A)	1.02(2)	C(15)-N(1)-C(18)	120.71(16)
C(3)-H(3B)	1.04(3)	C(3)-C(1)-C(2)	112.8(2)
C(3)-H(3C)	1.00(2)	C(3)-C(1)-S(1)	110.79(17)
C(4)-C(5)	1.526(3)	C(2)-C(1)-S(1)	108.47(17)
C(5)-C(6)	1.537(3)	C(3)-C(1)-H(1)	113.1(12)
C(5)-C(7)	1.555(3)	C(2)-C(1)-H(1)	106.6(12)
C(5)-H(5)	0.91(2)	S(1)-C(1)-H(1)	104.6(10)
C(6)-H(6A)	1.04(2)	C(1)-C(2)-H(2A)	112.7(15)
C(6)-H(6B)	0.99(2)	C(1)-C(2)-H(2B)	112.7(15)
C(6)-H(6C)	0.99(3)	H(2A)-C(2)-H(2B)	107(2)
C(7)-C(8)	1.520(3)	C(1)-C(2)-H(2C)	105.8(16)
C(7)-C(14)	1.545(3)	H(2A)-C(2)-H(2C)	106.0(19)
C(7)-H(7)	1.012(19)	H(2B)-C(2)-H(2C)	113(2)
C(8)-C(9)	1.397(3)	C(1)-C(3)-H(3A)	110.8(13)
C(8)-C(13)	1.399(3)	C(1)-C(3)-H(3B)	109.5(15)
C(9)-C(10)	1.388(3)	H(3A)-C(3)-H(3B)	106(2)
C(9)-H(9)	0.95(2)	C(1)-C(3)-H(3C)	110.1(13)
C(10)-C(11)	1.394(3)	H(3A)-C(3)-H(3C)	106.0(17)
C(10)-H(10)	0.97(2)	H(3B)-C(3)-H(3C)	114(2)
C(11)-C(12)	1.384(3)	O(1)-C(4)-C(5)	123.4(2)
C(11)-H(11)	1.02(2)	O(1)-C(4)-S(1)	123.78(16)
C(12)-C(13)	1.393(3)	C(5)-C(4)-S(1)	112.75(16)
C(12)-H(12)	0.97(2)	C(4)-C(5)-C(6)	107.83(18)
C(13)-H(13)	0.99(2)	C(4)-C(5)-C(7)	110.07(17)
C(14)-C(15)	1.512(3)	C(6)-C(5)-C(7)	112.63(18)
C(14)-H(14A)	0.974(19)	C(4)-C(5)-H(5)	109.3(14)
C(14)-H(14B)	0.94(2)	C(6)-C(5)-H(5)	111.9(15)
C(17)-C(18)	1.531(3)	C(7)-C(5)-H(5)	105.1(14)
C(17)-H(17A)	0.95(2)	C(5)-C(6)-H(6A)	110.8(14)
C(17)-H(17B)	0.96(2)	C(5)-C(6)-H(6B)	107.0(14)
C(18)-C(19)	1.541(3)	H(6A)-C(6)-H(6B)	109.8(17)
C(18)-H(18)	0.974(19)	C(5)-C(6)-H(6C)	110.8(14)
C(19)-C(20)	1.509(3)	H(6A)-C(6)-H(6C)	106.9(19)
C(19)-H(19A)	0.96(2)	H(6B)-C(6)-H(6C)	111(2)
C(19)-H(19B)	0.97(2)	C(8)-C(7)-C(14)	113.07(17)

C(8)-C(7)-C(5)	111.40(16)	O(4)-C(17)-H(17A)	108.8(13)
C(14)-C(7)-C(5)	108.83(17)	C(18)-C(17)-H(17A)	111.4(12)
C(8)-C(7)-H(7)	107.4(10)	O(4)-C(17)-H(17B)	111.0(11)
C(14)-C(7)-H(7)	109.0(11)	C(18)-C(17)-H(17B)	113.8(11)
C(5)-C(7)-H(7)	106.9(11)	H(17A)-C(17)-H(17B)	106.9(16)
C(9)-C(8)-C(13)	117.7(2)	N(1)-C(18)-C(17)	100.71(16)
C(9)-C(8)-C(7)	122.11(19)	N(1)-C(18)-C(19)	109.79(17)
C(13)-C(8)-C(7)	120.21(19)	C(17)-C(18)-C(19)	114.05(19)
C(10)-C(9)-C(8)	121.4(2)	N(1)-C(18)-H(18)	110.0(11)
C(10)-C(9)-H(9)	121.6(13)	C(17)-C(18)-H(18)	113.6(11)
C(8)-C(9)-H(9)	117.0(13)	C(19)-C(18)-H(18)	108.5(11)
C(9)-C(10)-C(11)	119.8(2)	C(20)-C(19)-C(18)	114.64(18)
C(9)-C(10)-H(10)	120.4(14)	C(20)-C(19)-H(19A)	114.0(11)
C(11)-C(10)-H(10)	119.7(14)	C(18)-C(19)-H(19A)	105.5(12)
C(12)-C(11)-C(10)	119.8(2)	C(20)-C(19)-H(19B)	109.0(12)
C(12)-C(11)-H(11)	119.8(12)	C(18)-C(19)-H(19B)	104.7(11)
C(10)-C(11)-H(11)	120.2(12)	H(19A)-C(19)-H(19B)	108.5(16)
C(11)-C(12)-C(13)	119.9(2)	C(21)-C(20)-C(25)	118.12(19)
C(11)-C(12)-H(12)	122.4(12)	C(21)-C(20)-C(19)	120.75(19)
C(13)-C(12)-H(12)	117.7(12)	C(25)-C(20)-C(19)	121.1(2)
C(12)-C(13)-C(8)	121.4(2)	C(22)-C(21)-C(20)	120.8(2)
C(12)-C(13)-H(13)	119.0(12)	C(22)-C(21)-H(21)	122.7(13)
C(8)-C(13)-H(13)	119.5(12)	C(20)-C(21)-H(21)	116.6(13)
C(15)-C(14)-C(7)	111.66(18)	C(23)-C(22)-C(21)	120.8(2)
C(15)-C(14)-H(14A)	110.9(10)	C(23)-C(22)-H(22)	119.5(13)
C(7)-C(14)-H(14A)	111.4(12)	C(21)-C(22)-H(22)	119.7(13)
C(15)-C(14)-H(14B)	106.6(11)	C(22)-C(23)-C(24)	119.1(2)
C(7)-C(14)-H(14B)	106.7(12)	C(22)-C(23)-H(23)	119.3(12)
H(14A)-C(14)-H(14B)	109.4(17)	C(24)-C(23)-H(23)	121.6(12)
O(2)-C(15)-N(1)	118.10(18)	C(23)-C(24)-C(25)	120.5(2)
O(2)-C(15)-C(14)	122.17(19)	C(23)-C(24)-H(24)	117.8(12)
N(1)-C(15)-C(14)	119.68(18)	C(25)-C(24)-H(24)	121.6(12)
O(3)-C(16)-O(4)	122.53(18)	C(24)-C(25)-C(20)	120.7(2)
O(3)-C(16)-N(1)	128.7(2)	C(24)-C(25)-H(25)	121.6(14)
O(4)-C(16)-N(1)	108.74(16)	C(20)-C(25)-H(25)	117.6(14)
O(4)-C(17)-C(18)	104.77(16)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for CJB12 (CCDC 239502). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	226(3)	252(3)	343(3)	-70(3)	131(2)	-41(3)
O(1)	262(8)	237(9)	360(10)	-68(7)	133(7)	-12(7)
O(2)	214(8)	303(10)	309(9)	-80(7)	81(7)	46(7)
O(3)	184(8)	228(9)	344(9)	-18(7)	97(7)	7(7)
O(4)	211(7)	223(9)	262(9)	-21(7)	107(6)	-19(7)
N(1)	128(8)	171(10)	220(10)	-16(7)	58(7)	-6(7)
C(1)	215(11)	274(13)	270(12)	-46(12)	115(10)	-51(12)
C(2)	347(15)	326(16)	439(17)	-32(13)	207(13)	-88(13)
C(3)	221(13)	489(19)	298(15)	1(13)	92(11)	17(13)
C(4)	149(10)	192(12)	248(12)	46(10)	31(9)	18(9)
C(5)	183(11)	180(12)	266(12)	15(11)	75(9)	15(10)
C(6)	249(12)	306(15)	332(14)	99(13)	88(11)	24(13)
C(7)	201(11)	152(12)	240(12)	-10(10)	104(9)	23(9)
C(8)	159(11)	165(11)	214(12)	25(9)	54(9)	19(9)
C(9)	239(12)	163(12)	335(13)	-59(11)	115(10)	-43(10)
C(10)	278(13)	297(15)	294(14)	-83(11)	145(11)	-50(11)
C(11)	220(11)	266(14)	299(13)	22(10)	110(10)	-68(10)
C(12)	266(13)	209(14)	286(14)	-32(11)	58(11)	-72(10)
C(13)	253(12)	195(12)	210(12)	1(10)	83(9)	10(10)
C(14)	205(12)	127(12)	239(13)	16(10)	77(10)	7(9)
C(15)	198(11)	149(11)	229(12)	13(9)	75(9)	-22(9)
C(16)	204(11)	90(11)	285(12)	5(10)	91(9)	-7(9)
C(17)	217(12)	231(13)	227(13)	-5(11)	48(10)	-13(11)
C(18)	190(11)	160(12)	239(12)	-4(10)	38(9)	7(10)
C(19)	186(11)	203(13)	248(13)	3(10)	72(9)	7(10)
C(20)	176(10)	211(13)	191(11)	21(9)	68(8)	-33(9)
C(21)	253(13)	191(12)	280(13)	-5(10)	116(11)	9(11)
C(22)	345(13)	241(13)	247(13)	-60(11)	127(11)	-94(12)
C(23)	221(12)	359(15)	229(13)	-14(11)	63(10)	-105(11)
C(24)	190(11)	297(15)	276(13)	16(11)	86(10)	-13(11)
C(25)	230(12)	199(13)	228(12)	-5(10)	92(10)	-16(10)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for CJB12 (CCDC 239502).

	x	y	z	U_{iso}
H(1)	-3984(15)	6140(30)	7273(14)	16(5)
H(2A)	-5220(20)	3220(40)	7012(16)	43(7)
H(2B)	-3970(20)	2490(40)	7070(19)	53(8)
H(2C)	-4950(20)	2890(40)	6019(19)	43(7)
H(3A)	-5515(16)	6420(40)	5367(16)	35(7)
H(3B)	-4960(20)	8460(50)	5996(19)	60(9)
H(3C)	-5881(19)	6860(40)	6297(15)	33(6)
H(5)	-1300(18)	7500(40)	5999(15)	31(7)
H(6A)	-2474(19)	10300(40)	5135(16)	40(7)
H(6B)	-1131(18)	10800(40)	5442(14)	35(6)
H(6C)	-1890(18)	11730(40)	6057(16)	34(7)
H(7)	-607(14)	9980(30)	7600(13)	14(5)
H(9)	641(17)	7380(40)	6020(15)	29(6)
H(10)	2089(17)	8770(40)	5494(15)	32(6)
H(11)	2939(16)	11990(30)	6123(14)	22(6)
H(12)	2189(16)	13800(40)	7213(14)	26(6)
H(13)	782(16)	12380(30)	7716(15)	23(6)
H(14A)	-737(15)	6430(30)	7985(12)	15(5)
H(14B)	-62(14)	5830(40)	7300(13)	15(5)
H(17A)	1882(16)	7250(40)	11317(15)	26(6)
H(17B)	2120(14)	9590(30)	11394(13)	10(5)
H(18)	2598(15)	7240(30)	10078(13)	12(5)
H(19A)	2572(14)	10190(30)	9165(15)	20(6)
H(19B)	2024(16)	11440(30)	9809(13)	13(5)
H(21)	3151(16)	13730(40)	10852(13)	19(5)
H(22)	4888(16)	14780(30)	11864(15)	21(6)
H(23)	6462(17)	12780(30)	12050(14)	20(6)
H(24)	6276(17)	9560(30)	11293(13)	23(6)
H(25)	4517(17)	8500(40)	10289(15)	34(7)