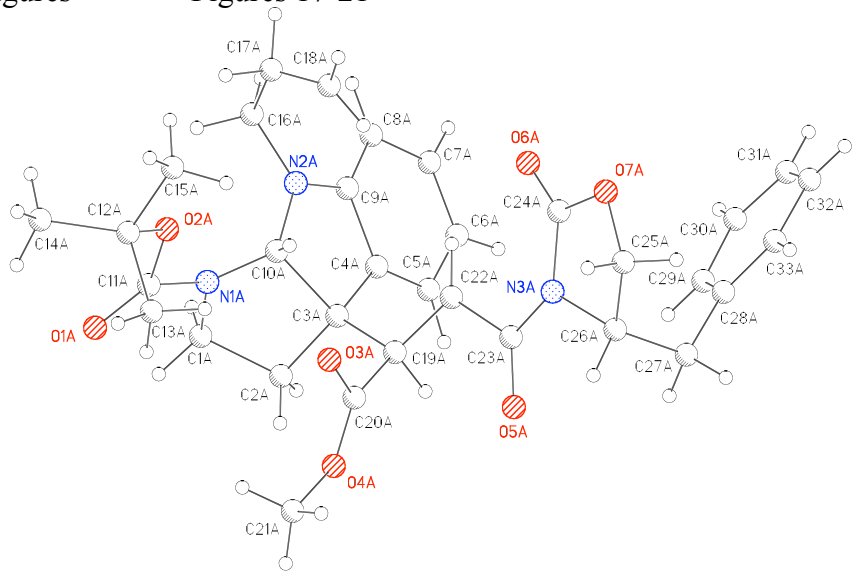
X-Ray Crystallographic data for JFA03

Table 11. Crystal data
Figures Figures 17-21



JFA03

Note: CCDC 234570 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033. Structure factors are available from the authors via e-mail: xray@caltech.edu

Table 11. Crystal data and structure refinement for JFA03 (CCDC 234570)

Empirical formula	$C_{33}H_{39}N_3O_7$
Formula weight	589.67
Crystallization solvent	Benzene/Hexane
Crystal habit	Blade
Crystal size	0.30 x 0.08 x 0.04 mm^3
Crystal color	Colorless

Data collection

Preliminary photos	Rotation	
Type of diffractometer	Bruker smart 1000	
Wavelength	0.71073 Å MoKα	
Data collection temperature	100(2) K	
θ range for 1808 reflections used		
in lattice determination	2.31 to 29.95°	
Unit cell dimensions	a = 8.911(3) Å	
	b = 35.439(11) Å	β= 92.251(6)°
	c = 9.979(3) Å	
Volume	3149.0(17) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	$P2_1$	
Density (calculated)	1.244 g/cm^3	
F(000)	1,256	
Data collection program	Bruker smart v5.054	
θ range for data collection	2.04 to 28.40°	
Completeness to θ = 28.40°	89.8%	
Index ranges	$-11 < h < 11$, $-31 < k < 45$, $-12 < l < 13$	
Data collection scan type	ω scans at 3 ϕ settings	
Data reduction program	Bruker saint v6.45	
Reflections collected	19,204	
Independent reflections	9,952 [R_{int}= 0.2073]	
Absorption coefficient	0.088 mm^{-1}	
Absorption correction	None	
Maximum and minimum transmission	0.9965 and 0.9742	

Table 11 (cont.)

Structure solution and refinement

Structure solution program	shelxs-97
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	shelxl -97
Refinement method	Full matrix least-squares on F^2
Data/restraints/parameters	9,952/1/353
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.511
Final *R* indices [I>2σ(I), 3250 reflections]	$R^1 = 0.1539$, $wR^2 = 0.2733$
R indices (all data)	$R^1 = 0.3072$, $wR^2 = 0.2968$
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Maximum shift/error	0.004
Average shift/error	0.000
Largest difference peak and hole	0.821 and -0.567 e.Å^{-3}

Special Refinement Details

The crystals were of low quality and diffracted poorly. As shown in Table 12, the measured intensities represent only 90% of the possible measurements and less than one-third of those were stronger than two times their sigma. Consequently, it was not possible to obtain a satisfactory refinement of the structure. The results of this structure determination are useful only for the verification of relative stereochemistry.

Refinement of F^2 against all reflections. The weighted *R* factor (*wR*) 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.

All estimated standard deviations (esds) (except the esd in the dihedral angle between two least squares (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.