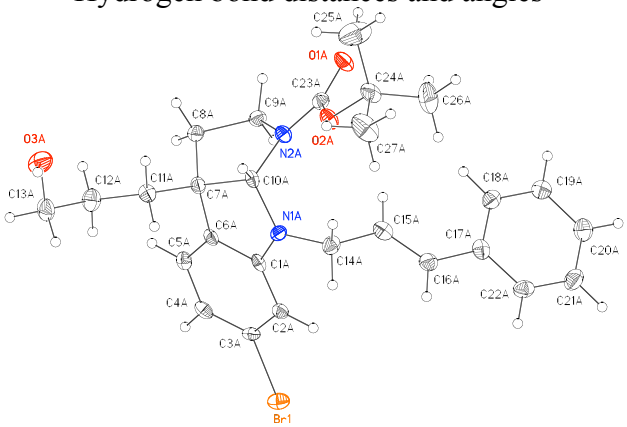
X-Ray Crystallographic Data for JFA01

Contents



JFA01

Note: CCDC 197024 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 4. Crystal data and structure refinement for JFA01 (CCDC 197024)

Empirical formula	$C_{27}H_{33}BrN_2O_3$
Formula weight	513.46
Crystallization solvent	Hexanes/Dichloromethane
Crystal habit	Block
Crystal size	0.17 x 0.22 x 0.22 mm^3
Crystal color	Colorless

Data collection

Preliminary photos	Rotation	
Type of diffractometer	Bruker smart 1000	
Wavelength	0.71073 Å MoKα	
Data collection temperature	98(2) K	
θ range for 18,881 reflections used in lattice determination	2.45 to 28.05°	
Unit cell dimensions	a = 8.8345(4) Å	
	b = 15.4918(7) Å	β= 97.229 (1)°
	c = 18.3034(9) Å	
Volume	2485.1(2) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	$P2_1$	
Density (calculated)	1.372 g/cm^3	
F(000)	1072	
Data collection program	Bruker smart v5.054	
θ range for data collection	1.73 to 28.37°	
Completeness to θ = 28.37°	95.2%	
Index ranges	$-11 \le h \le 11$, $-20 \le k \le 20$, $-24 \le l \le 24$	
Data collection scan type	ω scans at 7 ϕ settings	
Data reduction program	Bruker saint v6.022	
Reflections collected	51,296	
Independent reflections	11,520 [R_{int}= 0.0576]	
Absorption coefficient	1.685 mm^{-1}	
Absorption correction	None	

Table 4 (cont.)

Structure solution and refinement

Structure solution program	shelxs-97
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	shelxl-97
Refinement method	Full matrix least-squares on F^2
Data/restraints/parameters	11,520/1/603
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.255
Final R indices [I>2σ(I), 9,294 reflections]	$R^1 = 0.0330$, $wR^2 = 0.0561$
R indices (all data)	$R^1 = 0.0461$, $wR^2 = 0.0579$
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Maximum shift/error	0.002
Average shift/error	0.000
Largest difference peak and hole	0.984 and -0.316 e.Å^{-3}

Special Refinement Details

There are two molecules in the asymmetric unit. The conformation of each is very similar, with the exception of the propyl alcohol side group bonded to C7 (see Figure 7 and Table 6). Hydrogen bonds are formed between the hydroxyl group of this side group and the carbonyl oxygen of the group bonded to N2. All hydrogen atoms were restrained to ride on the atom to which they are bonded and the temperature factor set to 1.2 times the U_{eq} (1.5 times for methyl hydrogens) of the bonded atom. Hydroxyl hydrogens were allowed to rotate about the C-O bond and methyl hydrogens to rotate about the C-C to optimize the fit to electron density.

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.