Crystal structure of 2-acetyl-1-tribromomethyl-1,2,3,4-tetrahydro-β-carboline, $C_{11}H_{10}N_2(CBr_3)(COCH_3)$

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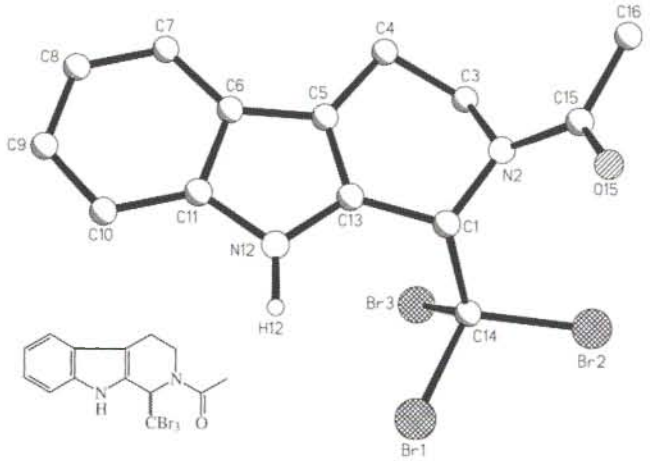

Abstract

$C_{14}H_{13}Br_3N_2O$, orthorhombic, *Pbca* (No. 61), $a = 14.165(1)$ Å, $b = 12.578(2)$ Å, $c = 16.923(2)$ Å, $V = 3015.1$ Å^3, $Z = 8$, $R_{gt}(F) = 0.083$, $wR(F) = 0.075$, $T = 293$ K.

Source of material

The title compound was prepared in 98% yield by *N*-acylation of 'TaBro' (1-tribromomethyl-1,2,3,4-tetrahydro-β-carboline), a potent neurotoxin for dopaminergic neurons [1, 2] with acetyl chloride in dichloromethane in the presence of triethylamine (3 h, room temperature).

Discussion

An intermolecular hydrogen bridge was found with *d*[H(12)···O(15)] = 214 pm to form a zigzag chain parallel [010].

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.55 × 0.6 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	80.20 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5581, 4109
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 2645
$N(param)_{refined}$:	181
Program:	SHELXTL-plus [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*c*	0.9363(6)	0.3478(8)	0.7238(5)	0.08
H(3A)	8*c*	0.7639(6)	0.2412(9)	0.5573(6)	0.08
H(3B)	8*c*	0.7250(6)	0.3364(9)	0.6074(6)	0.08
H(4A)	8*c*	0.7763(6)	0.4080(8)	0.4928(6)	0.08
H(4B)	8*c*	0.8720(6)	0.3458(8)	0.4975(6)	0.08
H(7)	8*c*	0.8665(6)	0.5946(9)	0.4306(6)	0.08
H(8)	8*c*	0.9159(6)	0.7698(9)	0.4260(6)	0.08
H(9)	8*c*	0.9734(6)	0.8551(9)	0.5372(7)	0.08
H(10)	8*c*	0.9837(6)	0.7674(8)	0.6584(7)	0.08
H(12)	8*c*	0.9471(5)	0.5700(6)	0.7321(5)	0.08
H(16A)	8*c*	0.8267(7)	0.1263(9)	0.5574(7)	0.08
H(16B)	8*c*	0.8603(7)	0.0368(9)	0.6154(7)	0.08
H(16C)	8*c*	0.9325(7)	0.0897(9)	0.5573(7)	0.08

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	8*c*	0.86839(8)	0.4623(1)	0.85375(7)	0.0813(7)	0.0734(8)	0.0346(6)	0.0028(6)	–0.0040(6)	–0.0044(6)
Br(2)	8*c*	0.77958(8)	0.2377(1)	0.81952(8)	0.0830(8)	0.0613(8)	0.0602(8)	0.0028(6)	0.0257(7)	0.0170(7)
Br(3)	8*c*	0.68806(7)	0.4446(1)	0.74965(9)	0.0568(6)	0.0850(9)	0.0576(7)	0.0206(6)	0.0045(6)	0.0066(8)
C(1)	8*c*	0.8765(6)	0.3634(8)	0.6997(5)	0.042(4)	0.051(6)	0.023(4)	0.001(4)	–0.004(4)	–0.008(5)
N(2)	8*c*	0.8501(5)	0.2785(7)	0.6487(5)	0.043(4)	0.048(5)	0.033(5)	–0.004(3)	–0.002(4)	0.010(4)
C(3)	8*c*	0.7806(6)	0.3053(9)	0.5847(6)	0.050(5)	0.057(7)	0.037(6)	–0.002(5)	–0.014(5)	–0.007(6)
C(4)	8*c*	0.8239(6)	0.3809(8)	0.5278(6)	0.051(6)	0.052(6)	0.043(7)	0.005(5)	–0.013(5)	–0.003(6)
C(5)	8*c*	0.8657(6)	0.4681(8)	0.5759(6)	0.035(4)	0.045(6)	0.041(6)	0.003(4)	–0.002(4)	0.000(5)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	8*c*	0.8940(5)	0.5718(8)	0.5487(6)	0.030(4)	0.055(6)	0.035(5)	0.005(4)	0.001(4)	0.003(5)
C(7)	8*c*	0.8896(6)	0.6288(9)	0.4775(6)	0.042(5)	0.068(7)	0.034(6)	0.004(5)	0.001(4)	0.008(6)
C(8)	8*c*	0.9194(6)	0.7314(9)	0.4749(6)	0.043(5)	0.069(8)	0.039(6)	0.000(5)	0.007(5)	0.016(6)
C(9)	8*c*	0.9537(6)	0.7823(9)	0.5413(7)	0.040(5)	0.055(7)	0.064(8)	–0.007(5)	–0.004(5)	0.018(6)
C(10)	8*c*	0.9601(6)	0.7315(8)	0.6125(7)	0.043(5)	0.052(7)	0.044(6)	–0.004(4)	–0.004(5)	–0.000(6)
C(11)	8*c*	0.9306(6)	0.6257(8)	0.6167(6)	0.030(4)	0.059(6)	0.039(6)	–0.002(4)	0.003(4)	0.003(5)
N(12)	8*c*	0.9266(5)	0.5554(6)	0.6791(5)	0.048(4)	0.048(5)	0.026(4)	–0.010(4)	–0.001(4)	0.002(4)
C(13)	8*c*	0.8879(6)	0.4618(9)	0.6540(6)	0.032(4)	0.068(7)	0.043(6)	–0.004(4)	–0.000(4)	0.005(6)
C(14)	8*c*	0.8055(7)	0.3746(9)	0.7764(6)	0.060(6)	0.052(6)	0.034(6)	0.004(5)	0.001(5)	0.015(5)
C(15)	8*c*	0.8999(6)	0.1842(8)	0.6512(6)	0.041(5)	0.053(6)	0.034(5)	0.002(4)	0.008(5)	0.001(5)
O(15)	8*c*	0.9628(4)	0.1707(6)	0.6993(4)	0.057(4)	0.060(5)	0.042(4)	0.013(3)	–0.000(4)	0.010(4)
C(16)	8*c*	0.8780(7)	0.1019(9)	0.5897(7)	0.069(6)	0.059(8)	0.044(7)	–0.007(5)	0.012(5)	–0.019(6)

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