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Structure of (*p*-Tolylcarbamoyl)(trimethylamino)borane

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Abstract. $C_{11}H_{19}BN_2O$, $M_r = 206·1$, orthorhombic, *Pbca*, $a = 20·102\,(3)$, $b = 12·607\,(4)$, $c = 9·981\,(2)$ Å, $V = 2529\,(1)$ Å^3, $Z = 8$, $D_x = 1·082$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1·54184$ Å, $\mu = 0·507$ mm^{-1}, $F(000) = 896$, $T = 223$ K. Of the 2401 unique reflections, 1785 were observed with $F_o \geq 6\sigma(F_o)$ giving $R = 0·042$. The molecule is in the elongated form, but is not planar; the torsion angle C(6)—C(1)—N(11)—C(11) = −27·7 (3)°. The bond distances N(11)—C(11) = 1·370 (2), B(11)—C(11) = 1·609 (2), B(11)—N(12) = 1·606 (2) Å. The molecules are linked by a weak hydrogen bridge N(11)—H(111)···O(11), N···O = 3·013 (2) Å.

Experimental. Colourless crystals from dichloromethane; crystal of approximate dimensions 0·50 × 0·45 × 0·05 mm; Cu $K\alpha$ radiation ($\lambda = 1·54184$ Å) was used with a graphite-crystal monochromator on a Nonius CAD-4 single-crystal diffractometer. Unit-cell dimensions from angular settings of 25 reflections with $14 < \theta < 27°$. The space group was determined from the systematic extinctions and the structure determination. The intensity data of 9213 reflections were measured (half a sphere up to $\theta = 58°$), using the ω–2θ scan technique, with a scan angle of 1·5° and a variable scan rate with a maximum scan time of 20 s per reflection. The maximum indices (*hkl*) were 24, 15 and 12, respectively. The intensity of the primary beam was checked by monitoring three standard reflections every 30 min. The final drift-correction factors were between 0·99 and 1·02. Profile analysis (Lehmann & Larsen, 1974; Grant & Gabe, 1978) was performed on all reflections; an empirical absorption correction was applied, using ψ scans (North, Philips & Mathews, 1968), $\mu = 5·07$ cm^{-1} (correction factors were in the range 0·79–1·00). Lorentz and polarization corrections were applied and the data were reduced to F_o values. Symmetry-equivalent reflections were averaged, $R_{\mathrm{int}} = \sum(|F_o - \langle F_o\rangle|)/\sum F_o = 0·038$ for all reflections and 0·017 for the observed reflections only, resulting in 2401 unique reflections of which 1785 were observed with $F_o > 6\sigma(F_o)$.

A *p*-aminotolyl skeleton of eight atoms was input to the vector-search rotation-function program *ORIENT* (Beurskens, Beurskens, Strumpel & Nordman, 1987); the oriented fragment was positioned by the reciprocal-space translation-function program *TRACOR* (Beurskens, Gould, Bruins Slot & Bosman, 1987) and automatically expanded by *DIRDIF* (Beurskens, Bosman, Doesburg, van den Hark, Prick, Noordik, Beurskens, Gould & Parthasathi, 1983). Isotropic least-squares refinement, using *SHELX* (Sheldrick, 1976) converged to $R = 0·14$. At this stage an additional empirical absorption correction was applied (Walker & Stuart, 1983), resulting

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Table 1. *Fractional positional and thermal parameters with e.s.d.'s in parentheses*

The expression for the equivalent isotropic vibrational parameter is: $U_{eq} = \frac{1}{3}\sum_i\sum_j a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j U_{ij}$.

	x	y	z	$100U_{eq}$ (Å^2)
C(1)	0·15783 (7)	0·30224 (12)	0·35280 (16)	2·98 (4)
C(2)	0·17951 (8)	0·37948 (13)	0·44244 (16)	3·30 (5)
C(3)	0·23923 (9)	0·43217 (14)	0·42203 (18)	3·87 (5)
C(4)	0·27871 (8)	0·41078 (15)	0·31062 (18)	4·05 (6)
C(5)	0·25626 (9)	0·33413 (16)	0·22203 (19)	4·47 (6)
C(6)	0·19698 (9)	0·28014 (16)	0·24153 (20)	4·22 (5)
C(11)	0·05306 (8)	0·20648 (13)	0·29075 (15)	3·09 (4)
C(13)	−0·02118 (11)	−0·00071 (17)	0·2199 (3)	5·71 (8)
C(14)	−0·11800 (11)	0·05570 (25)	0·34183 (25)	5·93 (8)
C(15)	−0·08422 (11)	0·15146 (22)	0·14423 (23)	5·56 (7)
C(41)	0·34377 (11)	0·46720 (24)	0·28686 (27)	6·08 (8)
B(11)	−0·01566 (11)	0·16927 (21)	0·35880 (21)	4·74 (7)
O(11)	0·06723 (5)	0·20171 (11)	0·16950 (11)	3·97 (4)
N(11)	0·09689 (6)	0·25077 (12)	0·37975 (14)	3·26 (4)
N(12)	−0·05968 (6)	0·09399 (11)	0·26397 (13)	3·44 (4)

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·392 (2)	C(11)—B(11)	1·609 (2)
C(1)—C(6)	1·389 (2)	C(11)—O(11)	1·245 (2)
C(1)—N(11)	1·412 (2)	C(11)—N(11)	1·370 (2)
C(2)—C(3)	1·387 (2)	C(13)—N(12)	1·489 (2)
C(3)—C(4)	1·392 (3)	C(14)—N(12)	1·487 (2)
C(4)—C(5)	1·385 (3)	C(15)—N(12)	1·482 (2)
C(4)—C(41)	1·508 (2)	B(11)—N(12)	1·606 (2)
C(5)—C(6)	1·386 (2)		

in a further decrease of R to 0·13. Relative absorption-correction factors were in the range 0·870–1·290.

The H atoms were found by difference Fourier synthesis techniques and included in the refinement with free atomic positions and isotropic temperature factors. During the final stages of the refinement all positional parameters and the anisotropic temperature factors of all non-H atoms were refined. The final conventional agreement factors were $R = 0{\cdot}042$, $wR = 0{\cdot}056$ and $S = 10{\cdot}67$ for 1785 'observed' reflections and 212 variables. The function minimized was $\sum w(F_o - |F_c|)^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}004F_o^2]$ and $\sigma(F_o)$ from counting statistics. The maximum shift-over-e.s.d. ratio in the last full-matrix least-squares cycle was less than 0·2. The final difference Fourier map showed maximum peaks at 0·18 e Å^{-3}. The scattering factors used were from *International Tables for X-ray Crystallography* (1974). Plots were made with *PLUTO* (Motherwell, 1976).

Final positional and thermal parameters are given in Table 1.* Bond lengths are collected in Table 2. A stereoview of the molecule, showing the molecular configuration, is given in Fig. 1. The crystallographic numbering scheme is shown in Fig. 2.

Related literature. Consideration of the isoelectric and isostructural character of CH_2 with BH_2^- units has led to the prediction and consequently the synthesis of a series of boron–nitrogen compounds of the general formula $R_3NBH_2CO_2H$ (R = H, alkyl or mixtures thereof), which may be considered as the boron analogues of α-amino acids in their dipolar forms (Spielvogel, Wojnowich, Das, McPhail & Hargrave, 1976; Spielvogel, Das, McPhail, Onan & Hall, 1980; Das & Mukherjee, 1985, 1987). Moreover, amide bonds are the principal linkages in polypeptides which are the essential constituents of all living systems. Therefore, attention has been drawn to the formation of such bonds between boron amino acids and amines (Spielvogel, Ahmed, Morse & McPhail, 1984; Das & Mukherjee, 1987) as the preliminary step towards the formation of polypeptides. On the basis of this premise, many boron analogues have been shown to be potential antineoplastic (Hall, Starnes, Spielvogel, Wisian-Neilson, Das & Wojnowich, 1979; Hall, Gilbert, McPhail, Morse, Hassett & Spielvogel, 1985; Sur, Roy & Das, 1981), hypolipidermic (Hall, Griffin, Docks, Brotherton & Futch, 1986; Hall, Spielvogel, Sood, Ahmed & Jafri, 1987) and anti-inflamatory (Hall *et al.*, 1987) agents. In view of the very low toxicity and very high inhibition against Ehrlich Ascites tumour growth of the amide $[CH_2N(CH_3)_2BH_2CONCH_2CH_3]_2$ (Hall *et al.*, 1985), we were tempted to synthesize a number of amides from both aromatic and aliphatic amines and study their structure–activity relationships. In this communication we report the structure of one such compound, namely, an aromatic amide of the boron amino acid. The B(11)—C(11) bond length of 1·609 (2) Å is sim-ilar to reported values (*e.g.* McPhail, Onan, Spielvogel & Wisian-Neilson, 1978).

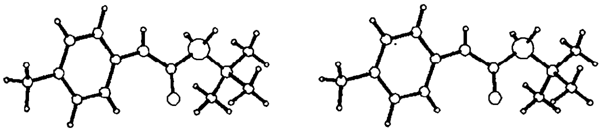

Fig. 1. Stereoview of the molecule showing the molecular configuration.

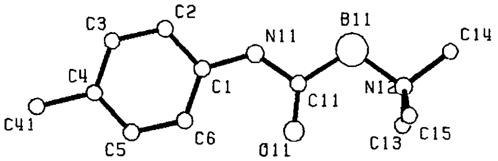

Fig. 2. Crystallographic numbering scheme. H atoms are numbered as the parent atom plus one additional digit (1, 2 or 3).

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms, positional and thermal parameters for H atoms, torsion angles, and geometrical and conformational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51874 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The B(11)—N(12) distance of 1·606 (2) Å is close to those in $Me_3NBH_2CO_2H$, 1·589 Å (Spielvogel *et al.*, 1976), and H_3NBH_2CN, 1·577 Å (McPhail *et al.*, 1978). The shortest intermolecular distance (excluding H atoms) is an N(11)···O(11) separation of 3·013 (2) Å, which is a weak hydrogen bond with an N(11)—H(111)···O(11) angle of 163 (2)°.

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Structure of an Enone Derivative

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Abstract. 2-Oxo-1,4,4-triphenyl-3-butenyl acetate, $C_{24}H_{20}O_3$, $M_r = 356{\cdot}42$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}717\,(1)$, $b = 21{\cdot}078\,(2)$, $c = 9{\cdot}054\,(2)$ Å, $V = 1854{\cdot}4(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}277\ \mathrm{g\,cm^{-3}}$, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 6{\cdot}00\ \mathrm{cm^{-1}}$, $F(000) = 752$, room temperature, $R = 0{\cdot}054$ for 1553 observed reflections $[F_o > 3\sigma(F_o)]$. There are no short intermolecular contacts less than the van der Waals contact distances. The phenyl groups have higher temperature factors than the ethylene group.

Experimental. Light-yellow crystals obtained from ethyl ether. Crystal of dimensions 0·2 × 0·2 × 0·2 mm rounded by a crystal grinder. Rigaku AFC-5 rotating-anode four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions determined from 20 2θ angles in the range $57 < 2\theta < 60°$. Intensities collected to $(\sin\theta)/\lambda = 0{\cdot}570$ Å^{-1} in h 0/11, k 0/25 and l 0/10, θ–2θ scans, θ-scan width $(1{\cdot}5 + 0{\cdot}15\tan\theta)°$, three standard reflections monitored every 100 reflections showed no significant variation in intensity. 1827 unique reflections measured, 1553 intensities observed $[F_o < 3\sigma(F_o)$ and two very strong reflections rejected], no absorption correction. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971). H atoms located on a difference map. Positional parameters of all atoms, anisotropic thermal parameters for C and O atoms, and isotropic thermal parameters for H atoms refined by block-diagonal least squares (Ashida, 1973). $\sum w|\Delta F|^2$ minimized with $w = 1{\cdot}0$ for $0 < F_o < 20$ and $w = [1{\cdot}0 + 0{\cdot}1(F_o - 20)]^{-1}$ for $F_o > 20$. Final

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