A single crystal investigation of L-tryptophan with *Z*' = 16

Carl Henrik Görbitz, Karl Wilhelm Törnroos and Graeme Day

Supplementary material

1. **Figure 1S** (below). Overlay of the eight molecules **A**, **B**, **E**, **F**, **I**, **J**, **M** and **N** of the *T* family (calculated by Sybyl-X, version 1.3; Tripos, 2011).

2. cif file for refinement model with extended constraints on anisotropic displacement parameters, 1338 refined parameters, final *R*-factor 0.0879.

3. *Acta Cryst.* C style preprint including complete listing of torsion angles and hydrogen bonding geometry.

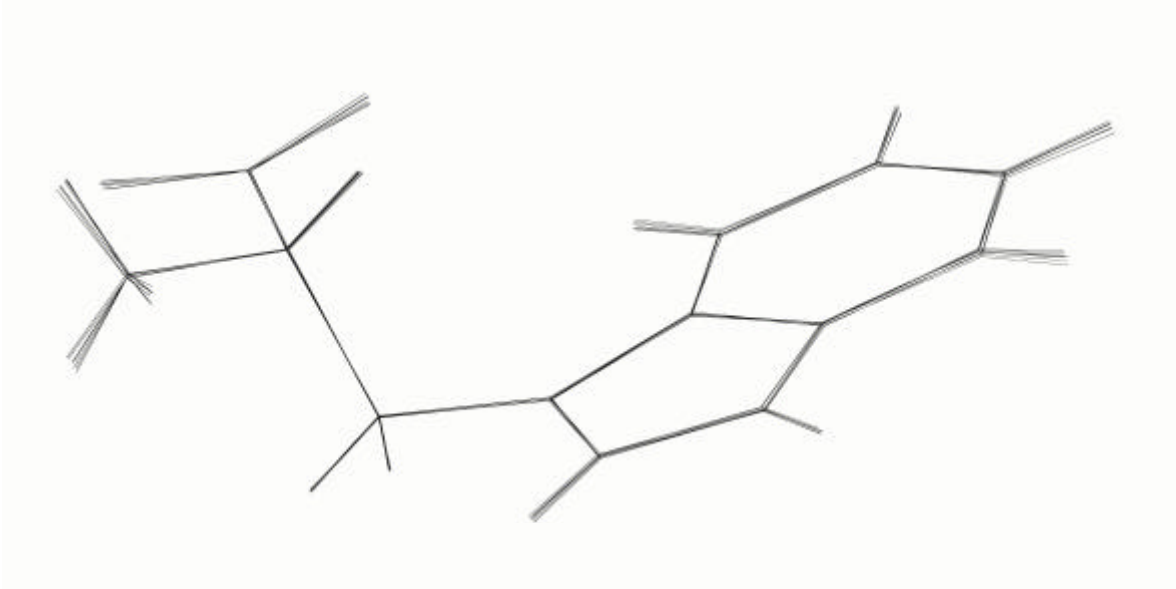

Figure 1S

Reference

Tripos (2011). *Sybyl-X 1.3*. Tripos International, St. Louis, Missouri, USA

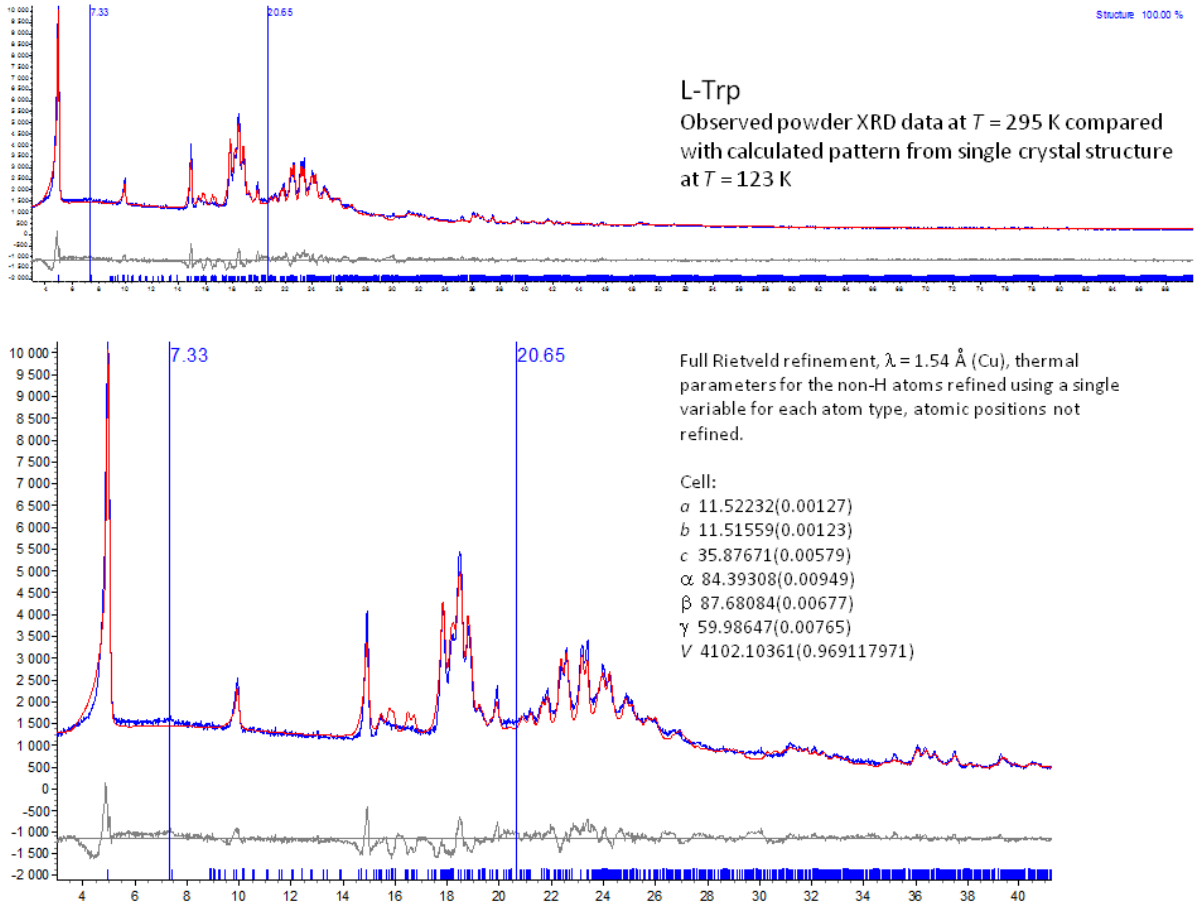

Figure 2S. Observed powder XRD data at T = 295 K compared with calculated pattern from single crystal structure at T = 123 K.

A single crystal investigation of L-tryptophan with $Z' = 16$

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A complex, disorder-free structure in space group *P*1 has been established for L-tryptophan, for which no crystal structure has previously been available. The 16 molecules in the asymmetric unit can be divided into two groups of eight molecules; one group where the side chains have *gauche*- orientations and one group with *trans* orientations. Molecules within each group have almost identical molecular geometries. The unit cell lengths mimic an hexagonal cell, but deviations from 90° for the cell angles α = 84.421 (4)° and β = 87.694 (4)° give a small tilt that rules out hexagonal (or trigonal) symmetry. The hydrogen bonding pattern resembles that found in the crystal structure of the racemic structure of DL-tryptophan, but the calculated density, hydrogen bond lengths and aromatic interactions show that the enantiomeric structure is less efficiently packed.

Comment

Text

Experimental

From a saturated solutions of L-Trp in water (approximately 10 µg/ml), 30 ml was deposited into a series of 30 x 6 mm test tubes. The tubes were subsequently sealed with parafilm. For each tube a needle was then used to prick a single small hole in the parafilm after which the tube was placed inside a larger test tube filled with 1 mL of acetonitrile. The system was ultimately capped and left for three days at 20 °C.

Crystal data

$C_{11}H_{12}N_2O_2$	γ = 60.102 (2)°
M_r = 204.23	V = 4025.6 (19) Å^3
Triclinic, *P*1	Z = 16
a = 11.430 (3) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
b = 11.464 (4) Å	μ = 0.10 mm^{-1}
c = 35.606 (9) Å	T = 123 K
α = 84.421 (4)°	0.62 × 0.28 × 0.14 mm
β = 87.694 (4)°	

Data collection

Bruker APEXII CCD diffractometer	24736 independent reflections
Absorption correction: Multi-scan (*SADABS*; Bruker, 2007)	19659 reflections with $I > 2\sigma(I)$
T_{min} = 0.943, T_{max} = 0.987	R_{int} = 0.043
66471 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.085	3 restraints
$wR(F^2)$ = 0.255	H-atom parameters constrained
S = 1.13	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
24736 reflections	$\Delta\rho_{min}$ = −0.47 e Å^{-3}
2178 parameters	

Table 1

Selected torsion angles (º)

N1A—C2A—C3A—C5A	−172.9 (4)	N1I—C2I—C3I—C5I	−173.1 (4)
C2A—C3A—C5A—C4A	−114.9 (6)	C2I—C3I—C5I—C4I	−113.3 (6)
N1B—C2B—C3B—C5B	−174.1 (4)	N1J—C2J—C3J—C5J	−173.6 (4)
C2B—C3B—C5B—C4B	−112.2 (6)	C2J—C3J—C5J—C4J	−113.2 (6)
N1C—C2C—C3C—C5C	−79.1 (5)	N1K—C2K—C3K—C5K	−78.2 (5)
C2C—C3C—C5C—C4C	111.2 (6)	C2K—C3K—C5K—C4K	109.8 (6)
N1D—C2D—C3D—C5D	−79.0 (5)	N1L—C2L—C3L—C5L	−79.8 (5)
C2D—C3D—C5D—C4D	112.1 (6)	C2L—C3L—C5L—C4L	113.5 (6)
N1E—C2E—C3E—C5E	−173.1 (4)	N1M—C2M—C3M—C5M	−173.4 (4)
C2E—C3E—C5E—C4E	−114.0 (6)	C2M—C3M—C5M—C4M	−112.6 (6)
N1F—C2F—C3F—C5F	−173.7 (4)	N1N—C2N—C3N—C5N	−173.4 (4)
C2F—C3F—C5F—C4F	−114.7 (6)	C2N—C3N—C5N—C4N	−115.2 (6)
N1G—C2G—C3G—C5G	−77.8 (5)	N1O—C2O—C3O—C5O	−78.7 (5)
C2G—C3G—C5G—C4G	109.6 (6)	C2O—C3O—C5O—C4O	111.8 (6)
N1H—C2H—C3H—C5H	−80.0 (5)	N1P—C2P—C3P—C5P	−79.1 (6)
C2H—C3H—C5H—C4H	113.6 (6)	C2P—C3P—C5P—C4P	111.3 (6)

Table 2

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O1*E*	0.91	1.91	2.797 (5)	165
N1*B*—H1*B*···O1*F*	0.91	1.90	2.784 (6)	165
N1*C*—H1*C*···O1*G*	0.91	1.98	2.820 (6)	152
N1*D*—H1*D*···O1*H*	0.91	1.94	2.813 (5)	159
N1*E*—H1*E*···O1*C*^{i}	0.91	1.94	2.789 (5)	155
N1*F*—H1*F*···O1*D*^{i}	0.91	1.93	2.798 (5)	159
N1*G*—H1*G*···O1*B*	0.91	1.95	2.811 (6)	158
N1*H*—H1*H*···O1*A*^{ii}	0.91	1.94	2.810 (5)	161
N1*I*—H1*I*···O1*M*	0.91	1.93	2.801 (5)	159
N1*J*—H1*J*···O1*N*	0.91	1.92	2.786 (6)	157
N1*K*—H1*K*···O1*O*	0.91	1.96	2.833 (5)	159
N1*L*—H1*L*···O1*P*	0.91	1.94	2.812 (5)	161
N1*M*—H1*M*···O1*L*^{iii}	0.91	1.95	2.796 (5)	155
N1*N*—H1*N*···O1*K*	0.91	1.91	2.789 (5)	162
N1*O*—H1*O*···O1*I*	0.91	1.94	2.813 (6)	160
N1*P*—H1*P*···O1*J*	0.91	1.96	2.809 (6)	154
N1*A*—H2*A*···O2*B*	0.91	1.83	2.704 (6)	161
N1*B*—H2*B*···O2*A*^{ii}	0.91	1.84	2.716 (6)	162
N1*C*—H2*C*···O2*D*	0.91	1.81	2.716 (6)	176
N1*D*—H2*D*···O2*C*^{ii}	0.91	1.83	2.713 (6)	165
N1*E*—H2*E*···O2*F*^{iv}	0.91	1.82	2.720 (6)	172
N1*F*—H2*F*···O2*E*	0.91	1.83	2.717 (6)	165
N1*G*—H2*G*···O2*H*^{iv}	0.91	1.79	2.697 (6)	172
N1*H*—H2*H*···O2*G*	0.91	1.81	2.709 (6)	167
N1*I*—H2*I*···O2*J*^{i}	0.91	1.80	2.699 (6)	169
N1*J*—H2*J*···O2*I*^{ii}	0.91	1.81	2.711 (6)	170
N1*K*—H2*K*···O2*L*^{i}	0.91	1.84	2.716 (6)	161
N1*L*—H2*L*···O2*K*^{ii}	0.91	1.83	2.713 (6)	162
N1*M*—H2*M*···O2*N*^{iv}	0.91	1.82	2.723 (6)	171
N1*N*—H2*N*···O2*M*^{v}	0.91	1.86	2.735 (6)	161
N1*O*—H2*O*···O2*P*^{iv}	0.91	1.79	2.692 (6)	169

N1*P*—H2*P*···O2*O*^{v}	0.91	1.79	2.699 (6)	177
N1*A*—H3*A*···O1*H*^{iv}	0.91	1.97	2.876 (6)	175
N1*B*—H3*B*···O1*G*	0.91	1.97	2.880 (6)	176
N1*C*—H3*C*···O1*E*^{v}	0.91	2.03	2.918 (6)	165
N1*D*—H3*D*···O1*F*^{v}	0.91	2.01	2.919 (6)	173
N1*E*—H3*E*···O1*A*	0.91	1.99	2.886 (6)	170
N1*F*—H3*F*···O1*B*	0.91	1.99	2.895 (6)	175
N1*G*—H3*G*···O1*C*	0.91	2.02	2.916 (6)	166
N1*H*—H3*H*···O1*D*	0.91	2.03	2.926 (6)	167
N1*I*—H3*I*···O1*O*	0.91	1.97	2.870 (6)	172
N1*J*—H3*J*···O1*P*	0.91	1.97	2.875 (6)	171
N1*K*—H3*K*···O1*N*	0.91	2.01	2.915 (5)	175
N1*L*—H3*L*···O1*M*^{vi}	0.91	2.02	2.928 (6)	173
N1*M*—H3*M*···O1*I*	0.91	1.99	2.891 (6)	170
N1*N*—H3*N*···O1*J*	0.91	1.98	2.893 (5)	179
N1*O*—H3*O*···O1*K*	0.91	2.03	2.925 (6)	166
N1*P*—H3*P*···O1*L*	0.91	2.04	2.919 (6)	161

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*, *y*+1, *z*; (iii) *x*−1, *y*−1, *z*; (iv) *x*, *y*−1, *z*; (v) *x*+1, *y*, *z*; (vi) *x*+1, *y*+1, *z*.

The structure of was refined without constraints or restraints on C, N or O positions. No postional disorder was indicated. H atoms were positioned with idealized geometry with fixed N—H = 0.88 (aromatic) or 0.91 Å (amino) and C—H = 0.95 (aromatic), 0.99 (methylene) or 1.00 Å (methine), while permitting free rotation for the amino groups. U_{iso} values were set to 1.2U_{eq} of the carrier atom, or 1.5U_{eq} for amino groups. In the absence of significant anomalous scattering effects, 23163 Friedel pairs were merged.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(type here to add acknowledgements)

References

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Figure 1

The paper contains seven figures and two schemes