The Crystal Structure at 105 K of Two Compounds Derived from Tryptamine, 1-Trifluoroacetyl-3-(2-trifluoroacetamidoethyl)indole and Tryptamine 3,4-Dimethoxybenzoate[§]

Sutatip Siripaisarnpipat* and Sine Larsen

Department of Physical Chemistry, H. C. Ørsted Institute, University of Copenhagen, Universitetsparken 5, DK-2100 Copenhagen Ø, Denmark

Siripaisarnpipat, S. and Larsen, S., 1987. The Crystal Structure at 105 K of Two Compounds Derived from Tryptamine, 1-Trifluoroacetyl-3-(2-trifluoroacetamido-ethyl)indole and Tryptamine 3,4-Dimethoxybenzoate. – Acta Chem. Scand., Ser. A 41: 539–547.

The crystal structures of the two title compounds have been determined by X-ray diffraction methods. The crystals of compound 1, $C_{14}H_{10}N_2O_2F_6$, are monoclinic, space group $P2_1/c$, $Z = 4$, with $a = 8.503(2)$, $b = 10.115(4)$, $c = 16.639(3)$ Å and $\beta = 90.35(2)°$. The structure of 1 is derived from tryptamine with the amino groups converted to amides of trifluoroacetic acid. There are significant differences between the geometries of the two amide groups. The bond lengths in the groups containing the imine nitrogen indicate that the bonds are more localized. This is also reflected in the geometry of the five-membered ring of the indole system. It exhibits variations that indicate that the bonds in this ring are more localized. Compound 2, $C_{19}H_{22}N_2O_4$, is a salt of tryptamine with 3,4-dimethoxy-benzoic acid; it crystallizes in the monoclinic space group $P2_1/c$ with $a = 14.479$ (6), $b = 6.733(4)$, $c = 18.609(6)$ Å, $\beta = 105.02(3)°$ and $Z = 4$. The conformation of the aminoethyl group in 2 differs from that observed previously in other tryptamine salts.

The indole ring system is widely distributed in biologically important compounds, mainly as tryptophan residues in proteins and as derivatives of tryptamine. A significant implication of the biological importance of tryptamine lies in its role as a starting material in the biosynthesis of alkaloids containing the indole ring system. This paper reports the determination of the crystal structures of two compounds obtained by two different reactions using tryptamine as a starting material.[1] The structure determinations were undertaken in order to unravel the identity of the reaction products, thereby providing additional

1

2
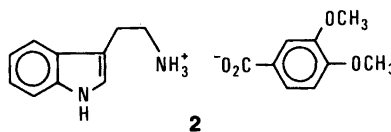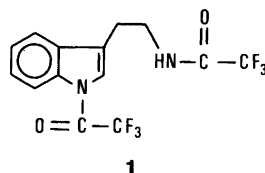
information about the structural chemistry of tryptamine. Compound 1 was obtained by reaction of tryptamine with trifluoroacetic acid anhydride. The second compound (2) is the salt of tryptamine and veratric acid, 3,4-dimethoxy-benzoic acid.

539

Table 1. Crystal data, and summary of data collection and structure refinements.

	1	**2**
Formula	$C_{14}H_{10}N_2O_2F_6$	$C_{19}H_{22}N_2O_4$
Formula weight	352.24	342.40
Unit cell parameters at ca. 105 K		
a/Å	8.503(2)	14.479(6)
b/Å	10.115(4)	6.733(4)
c/Å	16.639(3)	18.609(6)
β/deg.	90.35(2)	105.02(3)
V/Å^3	1430.9(10)	1752(2)
Space group	$P2_1/c$	$P2_1/c$
d_{exp}/g cm^{-3}	1.56	1.019
Z	4	4
Radiation (Mo$K\alpha$), λ/Å	0.71073	0.71073
Linear absorption coefficient, μ(Mo$K\alpha$)/cm^{-1}	1.547	0.857
Crystal size/mm	0.25×0.30×0.55	0.20×0.30×0.55
Scan mode	ω	ω-2θ
Scan range, $\Delta\omega$/°	1.5+0.35 tanθ	1.0+0.35 tanθ
θ-Range/°	1–35	1–30
Maximum scan time/s	150	150
Internal R-value from averaging	0.020	0.021
Number of independent reflections	6298	5108
Number of reflections, n, with $|F|^2 > 3\sigma(|F|^2)$	3715	2905
Number of variables, m	247	292
Weights, w^{-1}	$\sigma^2(F)+0.0009|F|^2$	$\sigma^2(F)+0.0016|F|^2$
R	0.042	0.041
R_w	0.055	0.055
Goodness of fit, $S = \dfrac{\Sigma w\Delta F^2}{n-m}$	1.336	1.102

Experimental

The compounds have been prepared by Prasit-pan.[1] To obtain crystals of a quality suitable for X-ray diffraction work, **1** was recrystallized from chloroform and **2** from dimethyl sulfoxide. The crystal data for the two compounds are listed in Table 1, which also contains a summary of the data collection and structure refinements. Additional details are presented in the following.

1-Trifluoroacetyl-3-(2-trifluoroacetamidoethyl)indole (**1**). This compound crystallizes as colourless parallelepipeds. Preliminary Weissenberg photographs showed that the crystals belong to the monoclinic system. The systematically absent reflections $h0l$ for $l = 2n+1$ and $0k0$ for $k = 2n+1$ are consistent only with the space group $P2_1/c$.

The density of the crystals was measured by flotation in an aqueous solution of KI. An Enraf-Nonius CAD 4 diffractometer equipped with a graphite monochromator and an Enraf-Nonius gas-flow low-temperature device was used for the data collection. The temperature was monitored by a thermocouple placed in the exhaust outlet a few cm above the crystal. The variation in the temperature was less than 1K. Accurate unit cell parameters were determined from a least-squares refinement of the setting angles for 18 reflections with $17 < \theta < 24°$. The scan type and scan range were selected on the basis of a detailed analysis of reflection profiles. The intensities of three standard reflections were measured after every 10 000 s, and the orientation of the crystal was checked after every 300 reflections by measuring setting angles for six reflections. These measure-

ments showed that no misalignment or deterioration of the crystal had occurred during the data collection. The data were corrected for Lorentz, polarization and background effects and the symmetry-related reflections were averaged.

Structure solution and refinement. The structure of **1** was solved by direct methods using MULTAN.[2] A standard run provided the positions of almost all of the non-hydrogen atoms in the structure. The remaining non-hydrogen atoms were found by Fourier syntheses. The structure was refined by the method of least-squares, minimizing $\Sigma w||F_o|-|F_c||^2$. A difference Fourier map calculated after an anisotropic refinement showed the positions of all the hydrogen atoms in the structure except one, which was introduced in a position calculated from idealized geometry. The positional parameters for the hydrogen atoms were also included in the refinement, a common fixed thermal parameter of $B = 2.00$ being used for all the hydrogen atoms. In the final refinement cycles the weights were changed from unit weights to weights of the form $w^{-1} = \sigma^2(F) + 0.0009|F|^2$. This scheme was selected as it gave a uniform distribution of $<w\Delta F^2>$ with $|F_o|$ and $\sin\theta/\lambda$. In the final refinement cycle the maximum shift was 0.22σ. The SDPVAX[3] system supplied by Enraf-Nonius was used for the crystallographic calculations. The atomic scattering factors given by Cromer and Mann,[4] and the anomalous dispersion corrections added to the scattering factors of F,O,C and N by Cromer and Liberman[5] were used as contained in the program system.

Tryptamine 3,4-dimethoxybenzoate (**2**). This compound crystallizes as pale yellow parallelepipeds. The crystallographic characterization was performed as described above for compound **1**. The density of the crystals was measured by flotation in a mixture of benzene and ethylene dibromide. Following the same procedure as described above the parameters for the data collection were selected. 25 reflections with $17<\theta<24°$ were used to determine the unit cell dimensions. Data col-

Table 2. 1-Trifluoroacetyl-3-(2-trifluoroacetamidoethyl)indole (**1**). Final fractional coordinates and values of B_{iso}.[a]

Atom	x	y	z	$B_{iso}/Å^2$
N1	0.3454(1)	0.6355(1)	0.42258(7)	1.06(2)
C2	0.3393(2)	0.4956(1)	0.41920(7)	0.99(2)
C3	0.3681(2)	0.4092(2)	0.35586(8)	1.29(2)
C4	0.3470(2)	0.2753(2)	0.37094(9)	1.56(2)
C5	0.2986(2)	0.2296(1)	0.44629(9)	1.61(2)
C6	0.2701(2)	0.3168(1)	0.50912(8)	1.33(2)
C7	0.2908(2)	0.4518(1)	0.49537(8)	0.99(2)
C8	0.2703(1)	0.5669(1)	0.54646(7)	0.97(2)
C9	0.3022(2)	0.6743(1)	0.50149(7)	1.04(2)
C10	0.2216(2)	0.5646(1)	0.63274(7)	1.11(2)
C11	0.0435(2)	0.5438(1)	0.63944(8)	1.17(2)
N12	−0.0083(1)	0.5462(1)	0.72305(6)	1.09(2)
C13	−0.0534(2)	0.4381(1)	0.76155(8)	1.16(2)
C14	−0.1185(2)	0.4613(1)	0.84641(8)	1.35(2)
F15	−0.1125(1)	0.5877(1)	0.86970(6)	2.22(2)
F16	−0.2684(1)	0.4234(1)	0.84979(6)	2.22(2)
F17	−0.0390(1)	0.3907(1)	0.90070(6)	2.26(2)
O18	−0.0520(1)	0.3245(1)	0.73580(7)	1.91(2)
C19	0.3942(2)	0.7151(1)	0.36137(8)	1.20(2)
C20	0.3703(2)	0.8654(1)	0.37420(8)	1.34(2)
F21	0.2221(1)	0.89400(9)	0.39415(6)	1.97(2)
F22	0.4642(1)	0.91212(9)	0.43259(6)	1.83(2)
F23	0.4032(1)	0.93050(9)	0.30762(6)	2.23(2)
O24	0.4513(1)	0.6768(1)	0.29924(6)	1.87(2)

[a]B_{iso} is calculated as $1/3\Sigma_i\Sigma_j B_{ij}a_i^*a_j^* \cdot a_i \cdot a_j^*$ for the atoms refined with anisotropic thermal parameters.

lection and processing were carried out as for **1**. Measurements of intensity and orientation on control reflections showed no systematic variations during the data collection.

Structure solution and refinement. The structure of **2** was solved and refined as described for **1**. The positions of all but one hydrogen atom were localized in a difference Fourier map, the remaining atom being introduced in an idealized position. All hydrogen atoms were given a fixed isotropic temperature factor, $B = 2.00$, and their positional parameters were included in the refinement. In the final refinement cycle the maximum shift was 0.07σ.

The positional parameters with equivalent isotropic temperature factors for **1** and **2** are listed in Tables 2 and 3, respectively. Anisotropic thermal parameters, parameters for the hydrogen atoms and listings of observed and calculated structure amplitudes can be obtained from the authors on request.

Description of the structures

1-Trifluoroacetyl-3-(2-trifluoroacetamidoethyl)indole (**1**). The molecular structure of this compound is illustrated by the ORTEP drawing in Fig. 1. The reaction with trifluoroacetic acid anhydride and tryptamine has lead to the formation of amides of both the amino and the imino group of tryptamine. The bond lengths and angles observed in **1** are listed in Table 4. The C–F bond lengths fall in the range 1.321–1.339 Å. These values agree well with the C–F bond lengths normally found in trifluoromethyl groups.[6-8] The two trifluoroacetyl groups exhibit systematic variations in the geometry of the N–C=O moieties. The C–N(amino) bond is significantly shorter (1.325 Å) than the C–N(imine) bond (1.365 Å), indicating a considerable amount of double bond character of the C–N(amine) bond. As expected, this increased double bond character of the C–N (amine) bond is accompanied by a lengthening of the C=O bond of the amine acetyl group [1.226

Table 3. Tryptamine 3,4-dimethoxybenzoate (**2**). Final fractional coordinates and values of B_{iso}.[a]

Atom	x	y	z	$B_{iso}/Å^2$
N1	0.6424(1)	0.2787(2)	−0.04862(7)	1.72(3)
C2	0.6793(1)	0.4579(3)	−0.01973(8)	1.28(3)
C3	0.7381(1)	0.5922(3)	−0.04401(9)	1.65(3)
C4	0.7658(1)	0.7615(3)	−0.0017(1)	1.77(3)
C5	0.7362(1)	0.7972(3)	0.0631(1)	1.68(3)
C6	0.6764(1)	0.6661(3)	0.08633(9)	1.37(3)
C7	0.6467(1)	0.4932(2)	0.04476(8)	1.11(3)
C8	0.5890(1)	0.3264(3)	0.05360(9)	1.25(3)
C9	0.5893(1)	0.1997(3)	−0.00395(9)	1.69(3)
C10	0.5440(1)	0.2935(3)	0.11700(8)	1.38(3)
C11	0.5920(1)	0.1194(3)	0.16400(9)	1.46(3)
N12	0.5647(1)	0.1025(2)	0.23591(7)	1.23(2)
C13	0.3204(1)	0.0797(2)	0.20242(8)	1.08(3)
C14	0.2149(1)	0.1114(2)	0.18853(8)	1.08(3)
C15	0.1517(1)	−0.0219(2)	0.14264(8)	1.15(3)
C16	0.0540(1)	0.0074(3)	0.12649(9)	1.35(3)
C17	0.0173(1)	0.1714(3)	0.15720(9)	1.39(3)
C18	0.0796(1)	0.3012(3)	0.20422(9)	1.41(3)
C19	0.1782(1)	0.2715(3)	0.21924(9)	1.32(3)
O20	0.37693(8)	0.2126(2)	0.23563(6)	1.44(2)
O21	0.34917(8)	−0.0816(2)	0.18041(6)	1.33(2)
O22	−0.01272(9)	−0.1112(2)	0.08133(7)	1.93(2)
O23	−0.07967(8)	0.1882(2)	0.13746(7)	1.95(2)
C24	0.0221(1)	−0.2745(3)	0.0466(1)	2.07(3)
C25	−0.1202(1)	0.3622(3)	0.1618(1)	2.30(4)

[a]Footnote as for Table 2.

Table 4. Bond lengths (Å), and bond and torsion angles (°) in **1**.

Bond lengths

N1–C2	1.418(2)		C19–O24	1.2092(14)
N1–C9	1.421(2)		C20–F21	1.337(2)
C2–C3	1.392(2)		C20–F22	1.339(2)
C3–C4	1.389(2)		C20–F23	1.3206(14)
C4–C5	1.401(2)		C10–C11	1.533(2)
C5–C6	1.390(2)		C11–N12	1.462(2)
C6–C7	1.397(2)		N12–C13	1.325(2)
C7–C2	1.407(2)		C13–C14	1.538(2)
C7–C8	1.452(2)		C13–O18	1.226(2)
C8–C9	1.348(2)		C14–F15	1.337(2)
C8–C10	1.497(2)		C14–F16	1.333(2)
N1–C19	1.365(2)		C14–F17	1.332(2)
C19–C20	1.548(2)			

Bond angles

C2–N1–C9	107.60(9)		C10–C11–N12	111.77(10)
C2–N1–C19	124.79(10)		C11–N12–C13	122.43(10)
C9–N1–C19	127.50(10)		N12–C13–C14	115.18(10)
N1–C2–C3	130.55(11)		N12–C13–O18	126.91(11)
N1–C2–C7	106.81(9)		C14–C13–O18	117.88(11)
C3–C2–C7	122.61(11)		C13–C14–F15	113.47(10)
C2–C3–C4	116.83(12)		C13–C14–F16	110.22(10)
C3–C4–C5	121.51(12)		C13–C14–F17	110.93(10)
C4–C5–C6	121.17(12)		F15–C14–F16	107.28(10)
C5–C6–C7	118.32(12)		F15–C14–F17	107.34(11)
C2–C7–C6	119.56(10)		F16–C14–F17	107.35(10)
C2–C7–C8	108.21(10)		N1–C19–O24	125.07(12)
C6–C7–C8	132.24(11)		N1–C19–C20	115.80(10)
C7–C8–C9	107.19(10)		C20–C19–O24	119.13(11)
C7–C8–C10	125.75(10)		C19–C20–F21	111.82(10)
C9–C8–C10	127.06(11)		C19–C20–F22	111.63(10)
N1–C9–C8	110.18(10)		C19–C20–F23	110.20(10)
C8–C10–C11	110.60(9)		F21–C20–F22	107.53(10)
			F22–C20–F23	107.69(10)
			F21–C20–F23	107.79(10)

Torsion angles

F15–C14–C13–N12	−3.0(2)		C9–C8–C10–C11	100.9(12)
F23–C20–C19–O24	6.9(2)		C8–C10–C11–N12	−177.84(11)
C14–C13–N12–C11	−174.54(11)		C10–C11–N12–C13	−107.70(14)
C20–C19–N1–C2	172.16(12)		C7–C8–C10–C11	−78.2(2)

(2) Å] compared to the acetyl group attached to the imine group [1.2092(14) Å]. The N(amino) acetyl group is very similar to that observed by Mostad and Rømming in the structure of melatonin.[9] Both trifluoromethyl groups adopt an eclipsed conformation relative to the N–C=O groups. In the N(amino) acetyl group this results in a short interatomic contact of 2.603(1) Å between F23 and O24, while in the other group the short contact is between F15 and N12 [2.636 (1) Å]. The six-membered ring of the indole system is strictly planar, whereas the five-membered ring is slightly puckered. The angle between the two least-squares planes is 1.3°.

The crystal packing of **1** is illustrated by the stereo pair shown in Fig. 2. As in the structure of melatonin,[9] the molecules are linked by hydrogen bonds between N12 and O18 from an adjacent
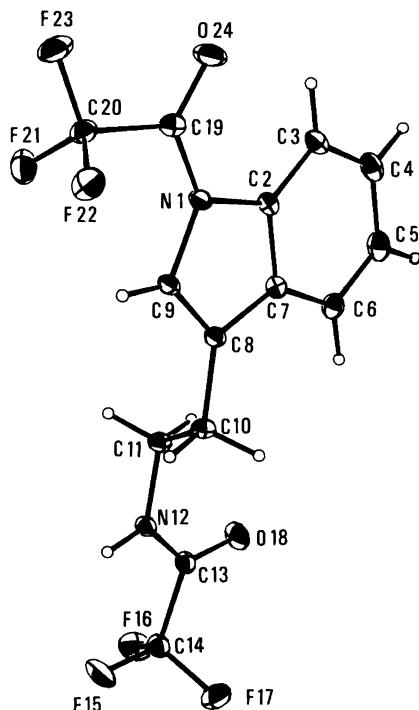
Fig. 1. An ORTEP drawing of **1** illustrating the atomic labelling. The thermal ellipsoids for the non-hydrogen atoms are scaled to include 50 % probability. The hydrogen atoms are drawn as spheres with an arbitrary radius.

molekyle related by the two-fold screw axis. The N12–O18 distance is 2.9412(14) Å and the N12–H12–O18 angle 157(2)°. There appear to be intermolecular interactions between the CF_3 groups. The shortest distance, viz. 2.8771(13) Å, is between F15 and F21. The other distances are slightly longer than twice the van der Waals radius for fluorine.[10]

Tryptamine 3,4-dimethoxybenzoate (**2**). This compound is a salt of tryptamine with 3,4-dimethoxybenzoic acid. Fig. 3 shows how the two ions are packed in the crystal, forming pairs linked by hydrogen bonds between the ammonium and the carboxylate groups. The N12–O20 distance is 2.817(2) Å and the N12–H12–O20 angle 157(3)°. The bond lengths, bond and torsion angles found in the anion have normal values and are listed in Table 5. As shown by the drawing in Fig. 4, the three substituents are almost coplanar with the benzene ring. The C16–O22 and C17–O23 bond lengths of 1.363(2) and 1.361 (2) Å, respectively, seem to confirm the observation made by Bryan and White[11] that the C17–O23 and C16–O22 bonds are significantly shorter than equivalent bonds in methoxy groups inclined to the plane of the phenyl group. The molecular dimensions of the tryptamine cation (shown in Fig. 5) are listed in Table 6. Salts of tryptamine with different acids have previously been characterized structurally,[12–15] and the bond lengths and angles found for the tryptamine cation in the present structure are in excellent
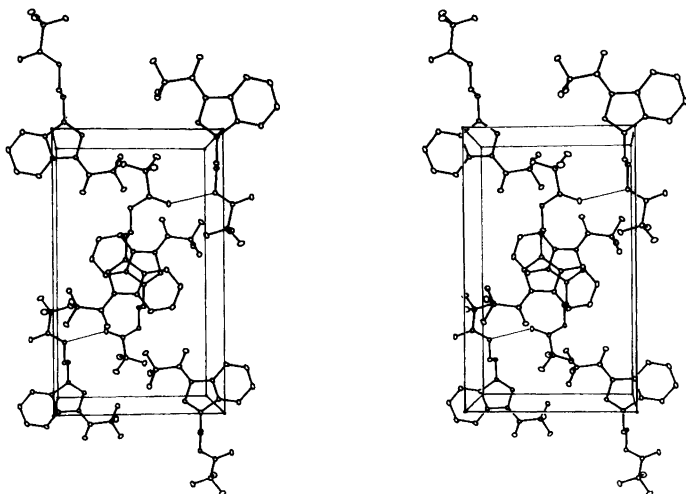

Fig. 2. A stereo pair illustrating the packing in **1** viewed along the cystallographic *a*-axis. The hydrogen bonds are shown as thin-line bonds.
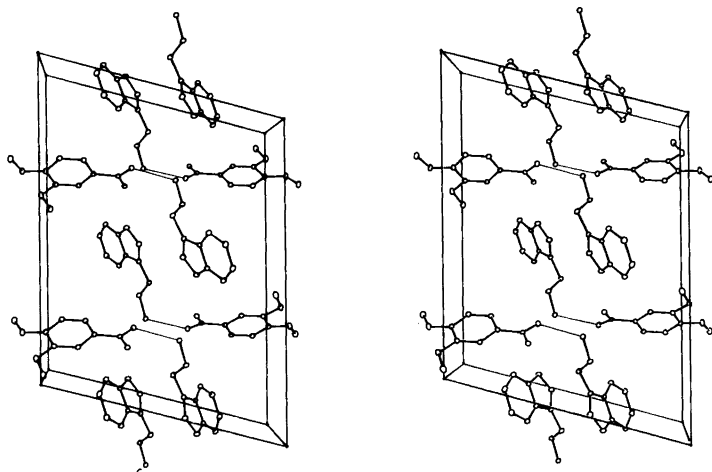
Fig. 3. The crystal packing in **2** seen along the crystallographic *b*-axis. The hydrogen bonds are indicated as thin-line bonds.

agreement with those observed in the other salts. Ishida *et al.*[14] compared the conformations found in the different salts. In the conformation observed more frequently, the torsion angle χ(C9–C8–C10–C11) is in the range 90–115° and the torsion angle φ(C8–C10–C11–N12) is in the (−) synclinical region with values ranging from −66 to −60°. The corresponding values found in **2** are χ = 63° and φ = 168.7°. In the structure of

tryptamine 1-thyminylacetate, Ishida *et al.*[14] found that the conformation of the aminoethyl group differs from the two previously described. This compound has χ = 4.2° and φ = 75.5°. In this context it should be mentioned that the number and strength of the hydrogen bonds involving the $-NH_3^+$ group are different in all tryptamine salts examined. Though Inoue *et al.*[16] found by molecular-orbital calculations that the most fre-

Table 5. Bond lengths (Å), and bond and torsion angles (°) in the 3,4-dimethoxybenzoate ion of **2**.

Bond lengths

C13–C14	1.498(2)	C17–C18	1.391(2)
C13–O20	1.261(2)	C18–C19	1.397(2)
C13–O21	1.268(2)	C16–O22	1.363(2)
C14–C15	1.402(2)	C17–O23	1.361(2)
C14–C19	1.388(2)	O22–C24	1.431(2)
C15–C16	1.381(2)	O23–C25	1.434(2)
C16–C17	1.409(2)		

Bond angles

O20–C13–C14	119.13(14)	C17–C16–O22	115.3(2)
O21–C13–C14	118.14(14)	C16–C17–C18	119.7(2)
O20–C13–O21	122.7(2)	C16–C17–O23	115.3(2)
C13–C14–C15	119.32(14)	C18–C17–O23	125.0(2)
C13–C14–C19	121.5(2)	C17–C18–C19	119.9(2)
C15–C14–C19	119.2(2)	C18–C19–C14	120.6(2)
C14–C15–C16	120.7(2)	C16–O22–C24	116.83(14)
C15–C16–C17	119.8(2)	C17–O23–C25	117.42(14)
C15–C16–O22	125.0(2)		

Torsion angles

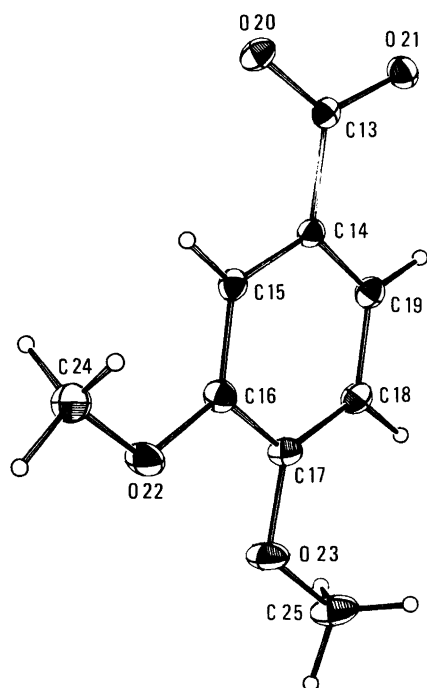|			
O20–C13–C14–C15	171.00(14)	C15–C16–O22–C24	−2.0(2)
		C18–C17–O23–C25	5.7(2)

Fig. 4. A perspective drawing of the
3,4-dimethoxybenxoate ion in **2**, drawn as described
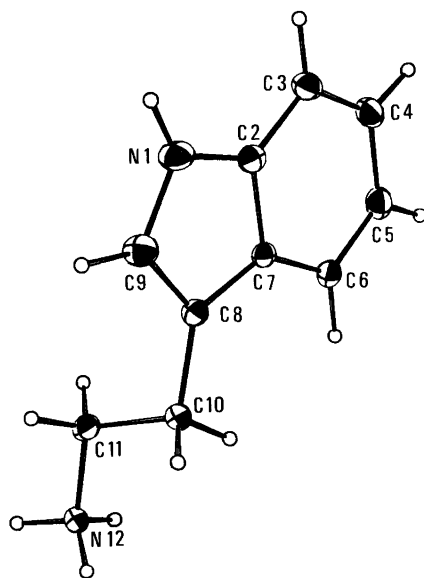for Fig. 1.

Fig. 5. The tryptamine cation in **2**, drawn as
described for Fig. 1.

Table 6. Bond lengths (Å), and bond and torsion angles (°) in the tryptamine cation of **2**.

Bond lengths

N1–C2	1.371(2)	C2–C7	1.419(2)
N1–C9	1.376(2)	C7–C8	1.434(2)
C2–C3	1.395(3)	C8–C9	1.370(2)
C3–C4	1.383(3)	C8–C10	1.504(2)
C4–C5	1.403(3)	C10–C11	1.518(2)
C5–C6	1.382(3)	C11–N12	1.494(2)
C6–C7	1.402(2)		

Bond angles

C2–N1–C9	108.92(14)	C2–C7–C8	107.30(14)
N1–C2–C7	107.3(2)	C6–C7–C8	133.7(2)
N1–C2–C3	130.7(2)	C7–C8–C9	106.09(14)
C3–C2–C7	122.0(2)	C7–C8–C10	126.1(2)
C2–C3–C4	117.5(2)	C9–C8–C10	127.7(2)
C3–C4–C5	121.5(2)	N1–C9–C8	110.3(2)
C4–C5–C6	121.0(2)	C8–C10–C11	109.82(13)
C5–C6–C7	119.0(2)	C10–C11–N12	112.77(14)
C6–C7–C2	119.0(2)		

Torsion angles

C9–C8–C10–C11	63.6(2)	C8–C10–C11–N12	168.70(13)
C7–C8–C10–C11	111.3(2)		

quently found conformation should be the most stable, it is apparent from the above that the intermolecular interactions in the crystal have a significant influence on the conformation of tryptamine.

Comparison of the two structures. The present structure determinations illustrate the effect of amide formation involving the imine nitrogen atom. In the indole ring system in **2**, the bonds in the five-membered ring have bond lengths corresponding to a delocalized system, as observed in other indole structures. In **1**, we find that the C8–C9 bond is shorter (1.348 Å compared with 1.370 Å) and that the N1–C9 and N1–C2 bonds are significantly longer than those in **2**. These systematic variations indicate that the amide formation at N1 leads to more localized bonds in the five-membered ring of the indole system.

References

1. Prasitpan, N. *To be published.*
2. Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J. P. and Woolfson, M. M. *MULTAN 80: A system of Computer Programs for the Automatic Solution of Structures from X-Ray Diffraction Data,* The Universities of York, England and Louvain, Belgium 1980.
3. *Structure Determination Package,* Enraf-Nonius, Delft, Holland 1981.
4. Cromer, D. T. and Mann, J. B. *Acta Crystallogr., Sect. A 24* (1968) 321.
5. Cromer, D. T. and Liberman, D. *J. Chem. Phys. 53* (1970) 1891.
6. Murthy, H. M. and Vijayan, M. *Acta Crystallogr., Sect. B 35* (1979) 262.
7. Cohen-Addad, C., Richard, J. M. and Guitel, J. C. *Acta Crystallogr., Sect. C 43* (1987) 504.
8. Mizuno, H., Kawashima, Y., Sota, K. and Kitamura, K. *Acta Crystallogr., Sect. C 43* (1987) 525.
9. Mostad, A. and Rømming, C. *Acta Chem. Scand., Ser. B 28* (1974) 564.
10. Bondi, A. *J. Phys. Chem. 68* (1964) 441.
11. Bryan, R. F. and White, D. H. *Acta Crystallogr., Sect. B 38* (1982) 1012.
12. Wakahara, A., Fujiwara, T. and Tomita, K. *Bull. Chem. Soc. Jpn. 46* (1973) 2481.
13. Gartland, G. L., Freeman, G. R. and Bugg, C. E. *Acta Crystallogr., Sect. B 30* (1974) 1841.
14. Ishida, T., Inoue, M. and Tomita, K. *Acta Crystallogr., Sect. B 35* (1979) 1642.
15. Inoue, M., Sakaki, T., Fujiwara, T. and Tomita, K. *Bull. Chem. Soc. Jpn. 51* (1978) 1123.
16. Inoue, M., Sakaki, T., Wakahara, A. and Tomita, K. *Biochim. Biophys. Acta 543* (1978) 123.