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2-(1-Methyl-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2] octan-3-one: Acid-catalyzed isomerization of the *Z* isomer to the *E* isomer

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Crystals of (*Z*)-2-(1-methyl-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2]octan-3-one (**I**) were obtained from a condensation reaction of 1-methyl-1H-indole-3-carboxaldehyde with 1-aza-bicyclo[2.2.2]octan-3-one and subsequent crystallization of the product from methanol. The isomeric (*E*)-2-(1-methyl-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2] octan-3-one hydrochloride (**II**) was obtained by treating a methanolic solution of **I** with a 1M solution of hydrogen chloride diethyl ether, followed by crystallization of resultant product from methanol. Crystal data: **I**, is monoclinic, $P2_1$, $a = 5.7440(10)$, $b = 11.102(2)$, $c = 10.708(2)$ Å, $\beta = 91.751(10)^\circ$, and $V = 682.5(2)$ Å^3 with $Z = 2$, for $D_{cal} = 1.296$ mg/m^3 and **II**, is monoclinic, $P2_1/c$, $a = 8.8510(2)$, $b = 17.4990(5)$, $c = 20.4300(5)$ Å, $\beta = 101.3620(12)^\circ$, $V = 3102.26(14)$ with $Z = 8$, for $D_{cal} = 1.316$ mg/m^3.

KEY WORDS: Heterocycle; indole; quinuclidinone; synthesis; single crystal x-ray structure.

Introduction

A variety of biological activities have been claimed for derivatives of indole. Recently, pyrido [1,2-*a*]indole derivatives have been identified as potent inhibitors of human immunodeficiency virus type 1,[1] and 5-chloro-3-(phenylsulfonyl) indole-2-carboxamide is reported to be a highly potent nonnucleoside inhibitor of HIV-1 reverse transcriptase.[2] Certain indole derivatives also exhibit antitumor activities.[3–7] We have shown that tryptamine analogs exhibit antagonist properties at the polyamine binding site on the *N*-methyl-D-aspartate (NMDA) receptor complex.[8] In the continuing search for novel tryptamine analogs with antagonist effects at the NMDA receptor, a series of 2-(*N*-substituted-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2]octan-3-ones were prepared. These indole analogs will be subjected to reductive deoxygenation to afford 2-(*N*-substituted-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2]octanes, which have been designed as rigid analogs of tryptamine. In the course of our studies, when a methanolic solution of (*Z*)-2-(1-methyl-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2]-octan-3-one was treated with an ethereal solution of hydrogen chloride, in order to prepare the more water-soluble hydrochloride salt, a facile isomerization to (*E*)-2-(1-methyl-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2]-octan-3-one hydrochloride in quantitative yield was observed,

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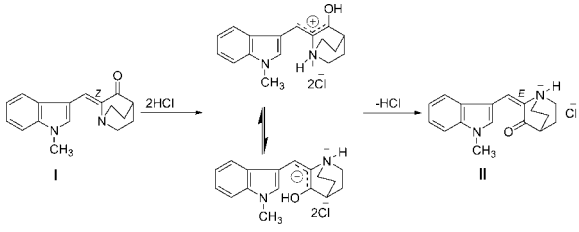
which was deduced from ^{1}H- and ^{13}C-NMR spectroscopy. We have confirmed this result from X-ray crystallographic analysis of the free base of **I**, and its hydrochloride salt, **II**. Confirmation of the double bond geometry in these molecules is important, since this geometry will be maintained in the subsequent deoxygenated final products, which we predict will be NMDA receptor ligands. We conducted a search on the isomerization of aza-bicyclo[2.2.2]octan-3-ones and found that Warawa *et al*. (1974) have reported an analogous isomerization of benzylidene-3-quinuclidinones with hydrogen chloride in chloroform,[9] and a mutual *Z*-/*E*-isomerization of ferromethylene- and arylidene-substituted carbo- and heterocycles has also been reported.[10] The possible mechanism by which this isomeric interconversion takes place is provided below.



Scheme I.

Experimental

All chemicals and solvents were of commercial reagent grade and were used without further purification.

(Z)-2-(1-Methyl-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2]octan-3-one (**I**)

To a stirred solution of diisopropylamine (1.923 g, 19 mmol) in THF (20 mL) at 0°C under nitrogen was added a solution of 2.0 M *n*-butyllithium (9 ml, 18.8 mmol) and the mixture stirred at 0°C for 30 min. To this solution at 0°C, was added 1-aza-bicyclo[2.2.2]octan-3-one hydrochloride (1.5 g, 9.28 mmol) in one portion and

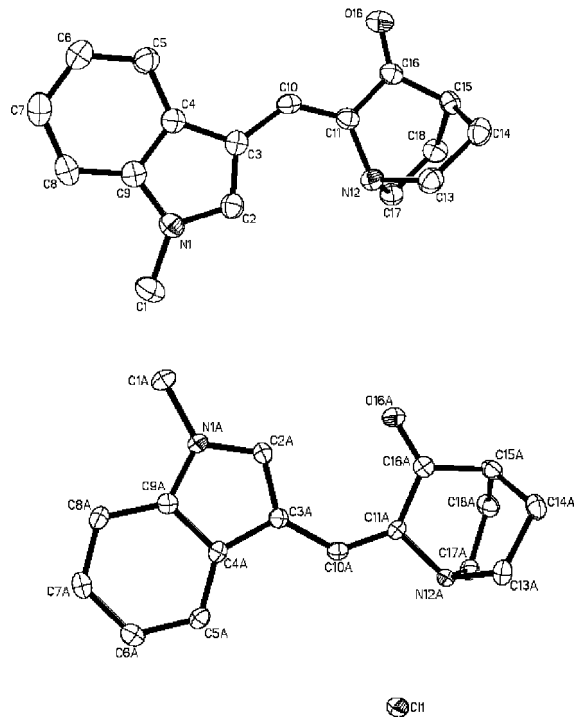


Fig. 1. Thermal ellipsoid plots of the molecules **I** and **IIA** with atom labeling (ellipsoids at 50% probability). Hydrogen atoms are omitted for clarity.

stirring continued until the mixture completely dissolved (20 min). The temperature was lowered to −78°C and a solution of 1-methyl-1H-indole-3-carboxaldehyde (1.46 g, 9.2 mmol) in THF (25 ml) was added drop wise. Stirring was continued for 30 min at this temperature and then at 0°C for 90 min. The reaction mixture was poured into saturated $NaHCO_3$ at 0°C and the resulting solution extracted with $CHCl_3$ (3 × 15 mL). The combined organic extracts were dried over anhydrous Na_2SO_4 and evaporated to afford a yellow solid. Crystallization from methanol afforded a yellow crystalline product, which was suitable for X-ray analysis; m.p. 170–172°C; ^{1}H NMR ($CDCl_3$, δ, ppm): 2.02 (4H, tb, J = 8.0 Hz, J′ = 3.0 Hz), 2.62 (1H, p, J = 3.0 Hz), 2.93–3.04 (2H, m), 3.11–3.21 (2H, m), 3.8 (3H, s), 7.19–7.36 (3H, m), 7.46 (1H, s), 7.9 (1H, d, J = 7.4 Hz), 8.28 (1H, s); ^{13}C NMR ($CDCl_3$, δ, ppm): 26.9, 33.6, 40.8, 47.9, 109.7, 109.9, 118.4, 119.1, 121.0, 122.6, 128.7, 134.9, 136.7, 140.4, 205.3.

Table 1. Crystal Data and Structure Refinement for **I**, and **II**

	I	**II**
Empirical formula	$C_{17}H_{18}N_2O$	$C_{17.25}H_{20}ClN_2O_{1.25}$
CCDC deposit no.	**215449**	**215448**
Formula weight	266.33	310.80
Temperature, K	173(1)	173(1)
Wavelength, Å	0.71073	0.71073
Crystal system,	Monoclinic	Monoclinic
Space group	$P2_1$	$P2_1/c$
Unit cell dimensions	$a = 5.7440(10)$ Å	$a = 8.8510(2)$ Å
	$b = 11.102(2)$ Å	$b = 17.4990(5)$ Å
	$c = 10.708(2)$ Å	$c = 20.4300(5)$ Å
	$\beta = 91.751(10)^\circ$	$\beta = 101.3620(12)^\circ$
Volume, Å^3	682.5(2)	3102.26(14)
Z	2	8
Density (calculated), mg/m^3	1.296	1.331
Absorption coefficient, mm^{-1}	0.082	0.250
$F(000)$	284	1316
Crystal size, mm	$0.34 \times 0.22 \times 0.20$	$0.35 \times 0.20 \times 0.10$
θ range for data collection, deg	2.64 to 27.48	2.03 to 25.00
Limiting indices	$-7 \le h \le 7$,	$-10 \le h \le 10$
	$-14 \le k \le 13$,	$-20 \le k \le 20$
	$-13 \le l \le 13$	$-24 \le l \le 24$
Reflections collected	4593	10740
Independent reflections	1644 [R(int) = 0.0454]	5470 [R(int) = 0.0325]
Data/restraints/parameters	1644/1/183	5470/30/400
Completeness to θ	= 27.48 99.8%	= 25.00 99.9%
Absorption correction	None	Multiscan
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Goodness of fit on F^2	1.039	1.112
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0462$, $wR_2 = 0.1092$	$R_1 = 0.0506$, $wR_2 = 0.0924$
R indices (all data)	$R_1 = 0.0715$, $wR_2 = 0.1223$	$R_1 = 0.0677$, $wR_2 = 0.0974$
Extinction parameter[14]	0.036(8)	0.0017(3)
Largest diff. Peak and hole, e/Å^{-3}	0.228 and −0.185	0.524 and −0.277

(E)-2-(1-Methyl-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2]octan-3-one hydrochloride (**II**)

To a solution of **I** (0.266 g, 1 mmol) in methanol (10 mL) at room temperature was added 1M hydrogen chloride in diethyl ether (2 mL) drop wise, and the mixture was stirred for 30 min. Evaporation of the solvents under vacuum afforded a yellow solid, which was crystallized from methanol; m.p. 228–230°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 2.03–2.16 (4H, m), 2.9 (1H, m), 3.35–3.40 (2H, m), 3.59-3.7 (2H, m), 3.9 (3H, s), 7.29–7.34 (2H, m), 7.60–7.63 (1H, m), 7.72–7.75 (1H, m), 8.35 (1H, s), 9.10 (1H, s), 13.3 (1H, b); ^{13}C NMR (DMSO-d_6, δ, ppm): 20.7, 33.7, 49.4, 51.1, 106.9, 111.3, 117.4, 121.9, 123.1, 126.0, 127.6, 136.7, 137.3, 193.2.

X-ray crystallography

X-ray diffraction data were collected at 90 K on a Nonius kappaCCD diffractometer from irregularly shaped crystals, mounted with oil on glass fibers. Initial cell parameters were obtained from ten 1° diffraction data frames and were refined via a least-squares scheme based on all frames (SCALEPACK, DENZO-SMN[11]). Lorentz/polarization corrections were applied during data reduction.

Table 2. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters (Å$^2 \times 10^3$) for **I**

Atom	*x*	*y*	*z*	*U*(eq)
N(1)	5324(4)	7413(3)	9960(2)	29(1)
C(1)	3718(6)	7074(3)	10931(3)	38(1)
C(2)	5301(5)	6947(3)	8777(3)	28(1)
C(3)	7067(5)	7462(3)	8107(3)	25(1)
C(4)	8231(5)	8307(3)	8943(3)	26(1)
C(5)	10137(5)	9077(3)	8833(3)	32(1)
C(6)	10820(6)	9771(3)	9849(3)	38(1)
C(7)	9666(6)	9712(3)	10974(3)	38(1)
C(8)	7783(6)	8944(3)	11111(3)	34(1)
C(9)	7087(5)	8250(3)	10087(3)	28(1)
C(10)	7635(5)	7284(3)	6819(3)	24(1)
C(11)	6854(5)	6449(3)	6003(3)	24(1)
N(12)	5314(4)	5473(2)	6307(2)	26(1)
C(13)	6413(5)	4318(3)	5935(3)	30(1)
C(14)	7036(5)	4310(3)	4546(3)	31(1)
C(15)	6231(5)	5508(3)	3945(2)	26(1)
O(16)	8672(3)	7272(2)	4192(2)	34(1)
C(16)	7436(5)	6503(3)	4664(3)	25(1)
C(17)	3128(5)	5603(3)	5551(3)	31(1)
C(18)	3592(5)	5641(3)	4127(3)	32(1)

Note. *U*(eq) is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

The structures were solved by direct methods and completed by difference Fourier techniques (SHELXS97, SHELXL97[12]). Refinement was carried out against F^2 by weighted full-matrix least-squares (SHELXL97[12]). Formal absorption corrections were not applied, but the frame-to-frame scaling in SCALEPACK[11] affords an approximate correction when anisotropic absorption is not too severe. All hydrogen atoms were found in difference maps and subsequently placed at calculated positions. They were refined using riding models with isotropic displacement parameters derived from their carrier atoms. Non-hydrogen atoms were refined with anisotropic displacement parameters. Atomic scattering factors were taken from the International Tables for Crystallography, Vol. C.[13] A molecule of solvent methanol was found to be disordered across an inversion centre in the crystal structure of **II**. This methanol was included in the model using half occupancy atoms that were restrained to chemically reasonable bonding parameters.[13]

Table 3. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters (Å$^2 \times 10^3$) for **IIA** and **IIB**

Atom	*x*	*y*	*z*	*U*(eq)
C(1A)	2492(3)	6686(2)	5580(1)	31(1)
C(2A)	2363(3)	5836(1)	4587(1)	20(1)
C(3A)	3351(3)	5522(1)	4209(1)	16(1)
C(4A)	4875(3)	5794(1)	4510(1)	16(1)
C(5A)	6344(3)	5690(1)	4371(1)	18(1)
C(6A)	7577(3)	6053(1)	4768(1)	22(1)
C(7A)	7381(3)	6513(1)	5305(1)	22(1)
C(8A)	5946(3)	6621(1)	5462(1)	20(1)
C(9A)	4710(3)	6255(1)	5056(1)	17(1)
C(10A)	3066(3)	5017(1)	3651(1)	17(1)
C(11A)	1756(3)	4721(1)	3284(1)	16(1)
N(12A)	1910(2)	4220(1)	2718(1)	16(1)
C(13A)	1131(3)	4589(1)	2076(1)	22(1)
C(14A)	−601(3)	4613(1)	2068(1)	24(1)
C(15A)	−906(3)	4391(1)	2756(1)	22(1)
O(16A)	−345(2)	5212(1)	3726(1)	27(1)
C(16A)	130(3)	4832(1)	3303(1)	19(1)
C(17A)	1201(3)	3444(1)	2769(1)	21(1)
C(18A)	−445(3)	3550(1)	2882(1)	26(1)
N(1A)	3156(2)	6265(1)	5088(1)	20(1)
C(1B)	2625(3)	1579(2)	2065(1)	26(1)
C(2B)	2879(3)	1548(1)	3306(1)	19(1)
C(3B)	1937(3)	1568(1)	3775(1)	17(1)
C(4B)	374(3)	1620(1)	3394(1)	17(1)
C(5B)	−1081(3)	1655(1)	3566(1)	20(1)
C(6B)	−2370(3)	1698(1)	3060(1)	23(1)
C(7B)	−2241(3)	1699(1)	2388(1)	22(1)
C(8B)	−825(3)	1662(1)	2202(1)	20(1)
C(9B)	468(3)	1623(1)	2715(1)	16(1)
C(10B)	2297(3)	1556(1)	4489(1)	18(1)
C(11B)	3636(3)	1485(1)	4936(1)	17(1)
N(12B)	3548(2)	1488(1)	5654(1)	19(1)
C(13B)	4547(3)	2111(1)	6024(1)	22(1)
C(14B)	6220(3)	1979(1)	5955(1)	24(1)
C(15B)	6300(3)	1262(1)	5531(1)	22(1)
O(16B)	5661(2)	1377(1)	4328(1)	31(1)
C(16B)	5232(3)	1378(1)	4863(1)	20(1)
C(17B)	4053(3)	726(1)	5962(1)	23(1)
C(18B)	5705(3)	576(1)	5874(1)	26(1)
N(1B)	2019(2)	1579(1)	2681(1)	18(1)
Cl(1)	5139(1)	3721(1)	2693(1)	36(1)
Cl(2)	360(1)	1959(1)	5851(1)	29(1)
O(1S)	300(20)	−90(9)	4851(4)	132(4)
C(1S)	1(16)	183(7)	5459(3)	70(3)

Note. *U*(eq) is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Results and discussion

Thermal ellipsoid plots for compound **I** and **IIA** are shown in Fig. 1. A summary of the crystal

Table 4. Selected Distances (Å) and Angles (deg) for **I**, **IIA**, and **IIB**

	I	IIA	IIB
N(1)—C(1)	1.461(4)	1.459(3)	1.463(3)
N(1)—C(2)	1.368(4)	1.349(3)	1.353(3)
C(2)—C(3)	1.384(4)	1.388(3)	1.389(3)
C(3)—C(10)	1.441(4)	1.425(3)	1.430(3)
C(10)—C(11)	1.341(4)	1.354(3)	1.351(3)
C(11)—N(12)	1.442(4)	1.480(3)	1.484(3)
C(11)—C(16)	1.483(4)	1.460(3)	1.461(3)
O(16)—C(16)	1.229(4)	1.228(3)	1.225(3)
N(12)—H(12)	—	0.9300	0.9300
C(2)—C(3)—C(10)	129.1(3)	131.2(2)	131.3(2)
C(11)—C(10)—C(3)	129.6(3)	132.6(2)	132.8(2)
C(10)—C(11)—C(16)	121.4(3)	132.5(2)	132.8(2)
C(10)—C(11)—N(12)	124.6(3)	117.3(2)	117.2(2)
C(16)—C(11)—N(12)	114.0(2)	110.04(19)	109.93(19)
C(11)—N(12)—C(17)	108.6(2)	112.08(17)	109.99(18)
C(13)—N(12)—C(17)	107.3(2)	108.75(17)	109.48(18)
N(12)—C(13)—C(14)	112.1(3)	107.97(19)	109.01(19)
C(13)—C(14)—C(15)	108.8(2)	109.85(19)	109.21(19)
C(16)—C(15)—C(18)	107.7(2)	105.29(19)	107.87(19)
C(14)—C(15)—C(18)	108.2(3)	107.8(2)	108.9(2)
O(16)—C(16)—C(11)	125.2(3)	124.5(2)	124.5(2)
O(16)—C(16)—C(15)	124.2(3)	123.7(2)	123.8(2)
C(11)—C(16)—C(15)	110.6(2)	111.8(2)	111.8(2)
N(12)—C(17)—C(18)	111.7(2)	108.86(19)	108.37(19)
C(17)—C(18)—C(15)	108.7(2)	108.7(2)	109.9(2)
C(2)—C(3)—C(10)—C(11)	−11.7(5)	5.0(4)	−3.7(5)
C(3)—C(10)—C(11)—C(16)	172.5(3)	3.1(5)	−1.4(5)

data are shown in Table 1, and atomic coordinates with equivalent isotropic displacement parameters are shown in Tables 2 and 3. Selected interatomic distances and bond angles are given in Table 4. In the asymmetric unit of compound **II**, there are two crystallographically independent molecules, and their geometries are nearly identical.

X-ray crystallography confirmed the molecular structures and atom connectivity for **I** and **IIA** as illustrated in Fig. 1. For each structure, the indole rings are planar with bond distances and angles comparable with those previously reported for other indole derivatives.[14,15] Chemically, both **I** and **II** represent the 1-aza-bicyclo[2.2.2]octan-3-one moiety substituted at position 2 with a 1-methyl-1H-indol-3-ylmethylene group. The *Z*-isomer, and the *E*-isomer hydrochloride, are characterized by a double bond between the C(10)—C(11) carbon atoms, which can assume the

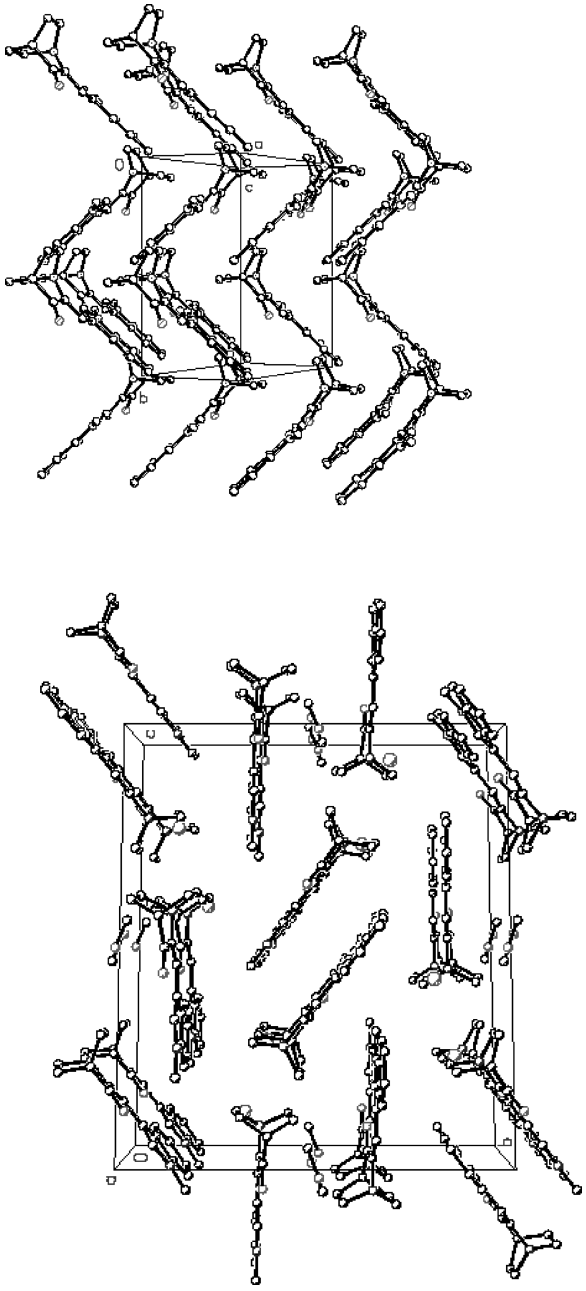


Fig. 2. Packing arrangements of the molecules **I** [viewed down the (1 0–1) axis] and **II** viewed down the *a* axis.

(*Z*) or the (*E*) geometrical disposition. The angles C(3)—C(10)—C(11), and C(10)—C(11)—N(12) in **I** are 129.6 (3)° and 124.6 (3)°, respectively, which are considerably larger than the ideal value of 120°, whereas the corresponding angles in

molecule **IIA/IIB** assume 132.6 (2)°/132.8 (2)°, and 117.3 (2)°/117.2 (2)° with simultaneous expansion and contraction of bond angles. However, the angle C(10)─C(11)─C(16) [121.4 (3)°] in **I** has ideal geometry, but in the molecule **IIA/IIB** it is larger 132.5 (2)°/132.8 (2)°. These deviations in molecule **I** and **IIA/IIB** are as a consequence of strain induced by the double-bond linkage at C(10)─C(11) connecting the 1-methyl-1H-indol-3-ylmethylene moiety with the 1-aza-bicyclo[2.2.2]octan-3-one ring and nonbonded interactions. In the case of the *E*-isomer hydrochloride, deviations are strong due to the additional nonbonded interaction between hydrogen atom at the C2 with carbonyl oxygen (O16)[2.89 Å]. The C3─C10 bond length in **I** [1.441 (4) Å], **IIA** [1.425 (3) Å], and **IIB**[1.430 (3) Å], when compared with the standard value for a single bond connecting a C_{ar} to a Csp^2 [1.470 ± 0.015 (4) Å] suggests extensive conjugation, beginning at the O16 carbonyl and extending through to the indole ring. The torsional angle [C2─C3─C10─C11] of 5.0 (4)°, and −3.7 (5)° for molecules **IIA**, and **IIB** suggests that the plane of the indole ring is almost planar compared to the plane of the double bond connected to the aza-bicyclic ring system, whereas in the case of molecule **I** there is only slight deviation [−11.7 (5)°].

The modes of packing of the *Z* isomer and *E* isomer hydrochloride of 2-(1-methyl-1H-indol-3-ylmethylene)-1-aza-bicyclo[2.2.2]octan-3-one are represented in Fig. 2. For the *Z*-isomer, van der Waal's forces are mainly responsible for the stabilization of the crystal. However, in the *E*-isomer hydrochloride crystal, where cocrystallized disordered methanol molecules and Cl^- ions are present in the unit cell together with the N-protonated quinuclidinone moiety, electrostatic interactions play a major role in the stabilization of the crystal. In both independent molecules protonated N atoms experience short interactions with Cl^- ions.

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