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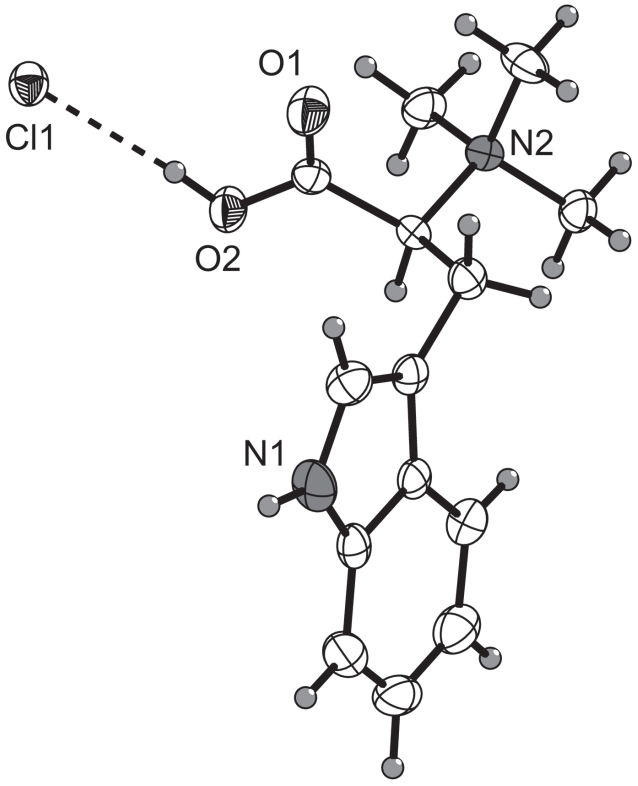
# The crystal structure of 1-carboxy-2-(1*H*-indol-3-yl)-*N*,*N*,*N*-trimethylethan-1-ammonium chloride, $C_{14}H_{19}N_2O_2Cl$

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## Abstract

$C_{14}H_{19}N_2O_2Cl$, tetragonal, $P4_1$ (no. 76), $a = 6.8614(8)$ Å, $c = 29.820(5)$ Å, $V = 1403.9(4)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0311$, $wR_{ref}(F^2) = 0.0549$, $T = 150(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | White polyhedron |
| Size: | 0.32 × 0.16 × 0.14 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.27 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker CCD, $\omega$ and $\varphi$-scans |
| $\theta_{max}$, completeness: | 27.0°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 11638, 3019, 0.038 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 2637 |
| $N(param)_{refined}$: | 183 |
| Programs: | Bruker [1], Olex2 [2, 3], SHELX [4] |

## Source of material

All reagents were purchased from Sigma-Aldrich and Merck Company. *Erythrina rubrinervia* Kunth seeds were collected on the Star farm, municipality of Villahermosa-Tolima, central Colombia, 5°02′40″ N, 75°7′38″ W, 1,860 m above sea level, by Olimpo García in February 2014. Prof. Alfredo Torres Benitez identified the plant material, and a voucher specimen was deposited in the Herbarium of the Biology Department Tolima of Tolima University UT12960. Extraction and Isolation: Dried and ground seeds (1.25 g) of *E. rubrinervia* were defatted with *n*-hexane and then exhaustively extracted with ethanol at room temperature over a period of 15 days. After concentration under reduced pressure, a viscous brown liquid (35.9 g) was obtained. Finally, after usual work-up for alkaloids, L-hypaphorine precipitated as a white solid (3.2 g).

## Experimental details

The structure was solved using OLEX2 [2] with the olex2.solve [3] and refined with the use of SHELX program package [4].

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.04018(10) | 1.45430(9) | 0.47638(2) | 0.02804(16) |
| O1 | 0.3779(3) | 1.1704(3) | 0.55229(7) | 0.0357(5) |
| O2 | 0.1235(3) | 1.0590(3) | 0.51304(7) | 0.0313(5) |
| H2O | 0.109(6) | 1.182(5) | 0.5081(13) | 0.090(15)* |
| N1 | −0.0824(4) | 0.8822(4) | 0.66525(9) | 0.0366(6) |
| H1N | −0.159(4) | 0.953(4) | 0.6790(10) | 0.045(10)* |
| N2 | 0.5147(3) | 0.7691(3) | 0.51914(7) | 0.0240(5) |
| C2 | 0.0943(4) | 0.9371(4) | 0.64793(10) | 0.0325(7) |
| H2 | 0.156505 | 1.054591 | 0.654017 | 0.039* |
| C3 | 0.1661(4) | 0.7953(4) | 0.62053(9) | 0.0238(6) |
| C4 | 0.0255(4) | 0.6407(4) | 0.62101(8) | 0.0239(6) |
| C5 | 0.0184(4) | 0.4546(4) | 0.60159(9) | 0.0304(7) |
| H5 | 0.117737 | 0.411349 | 0.582846 | 0.036* |
| C6 | −0.1392(5) | 0.3380(5) | 0.61102(10) | 0.0390(8) |
| H6 | −0.146392 | 0.215273 | 0.597905 | 0.047* |
| C7 | −0.2897(5) | 0.3979(5) | 0.63978(10) | 0.0428(8) |
| H7 | −0.393192 | 0.314328 | 0.645715 | 0.051* |
| C8 | −0.2848(4) | 0.5798(5) | 0.65925(10) | 0.0366(8) |
| H8 | −0.383377 | 0.621048 | 0.678422 | 0.044* |
| C9 | −0.1275(4) | 0.6994(4) | 0.64927(9) | 0.0258(6) |
| C10 | 0.3581(4) | 0.7932(4) | 0.59680(8) | 0.0267(7) |
| H10A | 0.422333 | 0.669379 | 0.602056 | 0.032* |
| H10B | 0.440574 | 0.895523 | 0.608809 | 0.032* |
| C11 | 0.3319(4) | 0.8239(4) | 0.54620(9) | 0.0203(6) |
| H11 | 0.222864 | 0.743159 | 0.535912 | 0.024* |
| C12 | 0.2824(4) | 1.0382(4) | 0.53742(9) | 0.0236(6) |
| C13 | 0.5472(4) | 0.5536(4) | 0.52253(9) | 0.0286(7) |
| H13A | 0.433201 | 0.486034 | 0.512224 | 0.043* |
| H13B | 0.572480 | 0.519242 | 0.553191 | 0.043* |
| H13C | 0.656892 | 0.517503 | 0.504341 | 0.043* |
| C14 | 0.6971(4) | 0.8725(4) | 0.53410(11) | 0.0337(7) |
| H14A | 0.804213 | 0.833677 | 0.515414 | 0.051* |
| H14B | 0.724882 | 0.838965 | 0.564699 | 0.051* |
| H14C | 0.678526 | 1.010802 | 0.531735 | 0.051* |
| C15 | 0.4800(4) | 0.8139(4) | 0.47083(9) | 0.0305(7) |
| H15A | 0.588874 | 0.769285 | 0.453399 | 0.046* |
| H15B | 0.465300 | 0.952054 | 0.467063 | 0.046* |
| H15C | 0.363669 | 0.749153 | 0.460957 | 0.046* |

H-atoms attached to the N1 and O2 atoms were located in the difference Fourier maps and their positions and isotropic displacement parameters were refined freely. All other H atoms were then treated as riding atoms in geometrically idealized positions.

## Comment

In this work, we report the isolation of L-hypaphorine from the seeds of *E. rubrinervia* and its crystallographic analysis. The genus Erythrina Mart. (Leguminoseae-Fabaceae) comprises around 115–118 known species [5–7] that grow in tropical-subtropical regions and in some temperate regions of the world in different ecosystems [7–9]. This alkaloid was first isolated from seeds of *Erythrina hypaphorus* Boerl [10]. However, it has been detected in several other genera and isolated from other species of Erythrina. The species of this genus are used traditionally to treat infections, such as malaria, inflammation, jaundice, anaemia, dysentery, female infertility, stomach pain, gonorrhea [11, 12] and for their anxiolitic effects. Moreover this compound has shown other interesting biological activities such as hypotensive, anticonvulsant, hypnotic, and analgesic ones [13–15].

The ORTEP diagram of the title structure with the atom-numbering scheme is shown in the figure. The indole group is essentially planar. The substituent group (at C3) is tilted out of the mean plane of the indole ring with a torsion angle C4—C3—C10—C11 of 76.1(3)°. The N1—C2 [1.371(4) Å] bond distance is similar to the average values reported for a Csp2-N in imidazole bond (1.370 Å) [16]. All the other relevant structural parameters (bond distances and angles) are as expected and in acceptable agreement with *L*-hypaphorine hydroiodide analogue [17]. In the crystal the molecules are linked *via* hydrogen bonds between chloride anions and organic molecules. The distance N1—H1N···Cl1 is 2.54(3) Å [angle of 157(3)°] and O2—H2O···Cl1 is 2.15(4) Å [angle of 162(4)°] and thus, the combination of both hydrogen bonds leads to the formation of chains running along the [001] direction.

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