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Stereochemistry of tropane alkaloid of convolvine and their derivatives

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KEYWORDS

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ABSTRACT

Structures of alkaloid convolvine (1) isolated from *Convolvulus subhirsutus* and its derivatives-convolamine(*N*-methylconvolvine) (2) and hydrochloride of *N*-benzylconvolvine (3) have been determined by single crystal X-ray diffraction technique. Compounds were crystallized in monoclinic space groups having four molecules in unit cell. All compounds contain a bicyclic ring system of tropane, where piperidine rings in all case adopt chair conformation. Hydrogen atom and methyl- and benzyl-substituents located in nitrogen atom of studied compounds occupy equatorial positions. The substituent of tropane core- the veratroyloxy group containing in all compound molecules is an α -axial oriented relative to the tropane core. In crystal structures of compound 1 and 2, the molecules are located in the distance of van der Waals interactions. The H-bond between the anion Cl and the proton of the N atom is observed in the crystal of *N*-benzylconvolvine hydrochloride (Cl···N 3.337 Å, Cl···H 2.42 Å and Cl-H-N 175°).

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1. Introduction

It is known that some tropane alkaloids (atropine, scopolamine) have a quinolytic effect and are used in medicine as drugs [1]. In order to search for physiologically active substances among alkaloids of this class, modification is carried out on the basis of available substances. So plants of the *Convolvulus* genus are a source of convolvine alkaloids, which can be produced on an industrial scale. Based on this alkaloid, we previously synthesized a number of derivatives [2]. Convolamine (2) was obtained by methylation of convolvine (1) and *N*-benzylconvolvine (3) was obtained by reaction of convolvine with benzyl chloride (Scheme 1). Biological studies on some cultures of cancer cells have established a high anticancer activity of benzylconvolvine, superior to the activity of anticancer drugs used in medicine [3].

The issue of obtaining convolvine derivatives is based on natural compound and it is dependent on the structure of the starting natural product and the reactant. These factors govern not only the derivation of new products, but also chemical property of product molecule, manifestation of biological activity by the formation of intra- and inter-molecular H- bonds, as well as the stereochemistry of the veratroyloxy group. In this connection, the stereochemical behavior of the nitrogen atom and substituent in derivatives is of interest. To this end, for the consideration of structural issues, a X-ray analysis on single crystals of the obtained derivatives was performed.



Scheme 1

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| Table 1. Crystal data and details of the structure refinement for compounds 1-3. | |
|--|--|
|--|--|

| Parameters | Compound 1 | Compound 2 | Compound 3 |
|-----------------------------------|---|---|-------------------------------------|
| Empirical formula | C ₁₆ H ₂₁ NO ₄ | C17H23NO4 | C23H28NO4·Cl |
| Formula weight | 291.34 | 305.36 | 417.91 |
| Temperature (K) | 293 | 293 | 293 |
| Crystal system | Monoclinic | Monoclinic | Monoclinic |
| Space group | Cc | $P2_1/n$ | $P2_1/c$ |
| a, (Å) | 10.2405(7) | 8.8351(5) | 15.4528(4) |
| b, (Å) | 20.429 (2) | 19.6231(11) | 9.0258(2) |
| c, (Å) | 7.1993(7) | 9.2514(5) | 16.5962(5) |
| β (°) | 94.587(7) | 91.977(5) | 111.121(3) |
| Volume (Å ³) | 1501.3(2) | 1603.0(2) | 2159.2(1) |
| Ζ | 4 | 4 | 4 |
| $\rho_{calc}(g/cm^3)$ | 1.289 | 1.265 | 1.286 |
| μ (mm ⁻¹) | 0.76 | 0.73 | 1.80 |
| F(000) | 624 | 656 | 888 |
| Crystal size (mm ³) | $0.60 \times 0.45 \times 0.40$ | 0.55 × 0.45 × 0.25 | $0.25 \times 0.30 \times 0.40$ |
| Radiation | CuKα (λ = 1.54184) | CuKα (λ = 1.54184) | $CuK\alpha$ ($\lambda = 1.54184$) |
| 20 range for data collection (°) | 9.6 to 152.6 | 9.0 to 152.6 | 10.8 to 176.4 |
| Index ranges | $-9 \le h \le 12$ | $-10 \le h \le 10$ | $-17 \le h \le 20$ |
| | $-22 \le k \le 25$ | $-17 \le k \le 24$ | $-11 \le k \le 8$ |
| | -9≤ <i>l</i> ≤ 8 | -11≤ <i>l</i> ≤11 | -19 ≤ <i>l</i> ≤ 18 |
| Reflections collected | 4762 | 6255 | 8728 |
| Independent reflections | 2197 [R _{int} = 0.045] | 3228 [R _{int} = 0.033] | 4317 [R _{int} = 0.033] |
| Data/restraints/parameters | 2197/2/193 | 3228/0/199 | 4317/0/269 |
| Goodness-of-fit on F ² | 1.067 | 0.996 | 1.022 |
| Final R indexes [I≥2σ (I)] | $R_1 = 0.061$, $wR_2 = 0.135$ | $R_1 = 0.051$, $wR_2 = 0.119$ | $R_1 = 0.050$, $wR_2 = 0.133$ |
| Final R indexes [all data] | R ₁ = 0.105, wR ₂ = 0.167 | R ₁ = 0.078, wR ₂ = 0.141 | $R_1 = 0.070$, $wR_2 = 0.146$ |
| Largest diff. peak/hole (e.Å-3) | 0.19/-0.15 | 0.16/-0.20 | 0.31/-0.22 |

2. Experimental

2.1. Materials and apparatuses

Convolvine (1) was isolated from *Convolvulus subhirsutus*. Reagents and solvents were purchased from commercial suppliers and used without further purifications. Single crystal X-ray diffraction (XRD) data was collected by using CuK α radiation ($\lambda = 1.54184$ Å) on a CCD Xcalibur Ruby diffractometer (Oxford Diffraction) at a room temperature. Data reduction including *multi-scan* absorption correction was done using *CrysAlisPRO* [4].

2.2. X-ray crystal structure determination of compounds 1-3

Structures were solved by direct methods within the SHELXS-97 program [5] and refined with SHELXL-2014/7 refinement program [6]. All non-hydrogen atoms were refined by the least squares method (F^2) in the full-matrix anisotropic approximation. Hydrogen atoms at carbon atoms were positioned geometrically and refined according to a riding model with fixed isotropic displacement parameters $U_{iso} = nU_{eq}$, where n = 1.5 for methyl groups and 1.2 for the others, (U_{eq} is the equivalent isotropic parameter of displacement of the corresponding carbon atoms). The hydrogen atoms of the NH group are found from difference syntheses of electron density and refined isotropically.

2.3. Preparation of convolvine and its derivatives

Isolation of convolvine (1) was performed according to the method [7]. The air-dried aerial part of *C. subhirsutus* (3 kg) was moistened with ammonia solution (10%), placed in a percolator, and after 2 h treated with CHCl₃ (six times). The combined CHCl₃ extracts were condensed to a volume of 2 L and worked up with H₂SO₄ solution (10%) to extract exhaustively the alkaloids. The combined acidic solutions were washed twice with a small quantity of CHCl₃ and made basic with ammonia solution (25%). The alkaloids were extracted with CHCl₃ to afford total bases (10.7 g). Total bases (10.7 g) were dissolved in CHCl₃ (2 L) and worked up with KOH solution (4%, 4 × 100 mL). The alkaline extracts were acidified with H₂SO₄ solution (20%), cooled, and made basic with

ammonia solution (25%). Alkaloids were extracted exhaustively with CHCl₃. The CHCl₃ solution was dried over anhydrous Na₂SO₄. Solvent was distilled off to produce phenolic alkaloids (2.5 g). After work up with base, the CHCl₃ solution was washed with distilled water and worked up successively with citrate-phosphate buffer at pH = 6.8 and 5.6 to extract completely the alkaloids. The buffer extracts were made basic with cooling using conc. NH₄OH solution. Alkaloids were extracted with CHCl₃ to afford bases from the fractions with pH = 6.8 (5.5 g, convolvine) and 5.6 (2.2 g, convolamine with an impurity of convolvine).

A mixture of 0.1 g of convolvine alkaloid, 1 mL of methyl iodide and 0.4 g of potassium iodide in 30 mL of dry acetone was heated in a water bath for 1 hour. When the cooled filtrate was concentrated, a crystalline precipitate (2) was formed with M.p.: 114-115 ° (acetone) in the amount of 0.11 g and not giving melting point depression with a convolamine sample.

Synthesis of *N*-benzylconvolvine (**3**) was carried out according to the method [2]. A mixture of convolvine (0.15 g) and benzylchloride (0.1 mL) was left at room temperature for 2 d. After this time the product was separated by treatment with acetone and purified over a column of Al_2O_3 to afford crystals (0.15 g, 78.9%) with M.p.: 88-89 °C.

3. Results and discussion

The molecular structures of convolvine **1** and its derivatives **2** and **3** are shown in Figure 1. Crystallographic data are presented in Table 1. The tropane alkaloid and derivatives are crystallized in the Cc space groups (containing glade plane) and $P2_1/n$, $P2_1/c$ (with elements of the center of symmetry and glade plane), respectively. Consequently, the crystals contain both enantiomers of the molecules of the alkaloid **1** and its derivatives **2** and **3**.

XRD analysis result allows to set the relative configuration of the center C3. The substituent the veratroyloxy group in position C3 has an α -axial orientation relative to the tropane core. The orientation and location of the substituents of the N and C3 atoms in compounds **1**, **2** and **3** coincides with those observed in convolinine [8] and *o*-benzoyltropine hydrochloride [9]. In molecules **1**-**3**, the veratroyloxy group is planar with an accuracy of ±0.040, ±0.039 and ±0.036 Å, respectively, and the benzyl group at N1 in **3** is ±0.008 Å.

| Fable 2. Selected bond lengths and bond angles of molecule 1. | | | | | |
|---|-----------------|----------------|-----------------|----------------|-----------------|
| Atom-Atom | Bond length (Å) | Atom-Atom | Bond length (Å) | Atom-Atom | Bond length (Å) |
| 01-C16 | 1.348 (8) | N1-C5 | 1.468 (10) | C6-C7 | 1.539 (11) |
| 01-C3 | 1.465 (7) | N1-H1A | 0.85 (8) | C1'-C6' | 1.379 (9) |
| 02-C16 | 1.205 (7) | C1-C2 | 1.517 (10) | C1'-C2' | 1.398 (8) |
| 03-C4' | 1.364 (7) | C1-C7 | 1.539 (9) | C1'-C16 | 1.486 (8) |
| 03-C17 | 1.427 (8) | C2-C3 | 1.513 (9) | C2'-C3' | 1.378 (8) |
| 04-C3' | 1.361 (7) | C3-C4 | 1.521 (10) | C3'-C4' | 1.413 (8) |
| 04-C18 | 1.424 (8) | C4-C5 | 1.534 (10) | C4'-C5' | 1.383 (8) |
| N1-C1 | 1.466 (10) | C5-C6 | 1.545 (10) | C5'-C6' | 1.379 (9) |
| Atom-Atom-Atom | Bond angles (°) | Atom-Atom-Atom | Bond angles (°) | Atom-Atom-Atom | Bond angles (°) |
| C16-01-C3 | 117.4 (5) | N1-C5-C4 | 106.6 (6) | 04-C3'-C4' | 114.6 (5) |
| C4'-03-C17 | 117.7 (5) | N1-C5-C6 | 106.3 (6) | C2'-C3'-C4' | 120.1 (5) |
| C3'-04-C18 | 116.8 (5) | C4-C5-C6 | 112.4 (6) | 03-C4'-C5' | 126.0 (5) |
| C1-N1-C5 | 100.9 (5) | C7-C6-C5 | 102.7 (6) | 03-C4'-C3' | 115.5 (5) |
| N1-C1-C2 | 106.8 (6) | C6-C7-C1 | 104.5 (6) | C5'-C4'-C3' | 118.5 (5) |
| N1-C1-C7 | 105.7 (6) | C6'-C1'-C2' | 120.5 (6) | C6'-C5'-C4' | 121.8 (6) |
| C2-C1-C7 | 112.7 (6) | C6'-C1'-C16 | 117.8 (5) | C5'-C6'-C1' | 119.3 (6) |
| C3-C2-C1 | 113.4 (6) | C2'-C1'-C16 | 121.7 (5) | 02-C16-O1 | 122.9 (6) |
| 01-C3-C2 | 109.2 (5) | C3'-C2'-C1' | 119.8 (6) | 02-C16-C1' | 124.5 (6) |
| 01-C3-C4 | 106.9 (5) | 04-C3'-C2' | 125.3 (6) | 01-C16-C1' | 112.6 (5) |



Figure 1. The molecular structures of compounds 1-3.

The veratroyloxy group is distorted from the plane of symmetry of the tropane core, which is characterized by the torsion angle H3-C3-O1-C1', whose values for compound **1-3** are 33, 29 and 33°, respectively. It should be noted that the carbonyl group in these compounds and analogues known in the literature [8-12] is always *syn*-directed relative to the β -

axially located hydrogen atom at C3. But the methoxyl group in the *ortho* position of the C3' veratroyloxy fragment in these compounds is located differently relative to the tropine core (Figure 1), which indicates a free rotation around the C16-C1' bond forming different rotamers. Table 3 Selected bond lengths and bond angles of molecule 2

| Atom-Atom | Bond length (A) | Atom-Atom | Bond length (Å) | Atom-Atom | Bond length (A) |
|--|---|--|---|--|--|
| 01-C16 | 1.340 (3) | N1-C5 | 1.469 (3) | C6-C7 | 1.536 (3) |
| 01-C3 | 1.468 (3) | N1-C1 | 1.474 (3) | C1'-C6' | 1.381 (3) |
| 02-C16 | 1.208 (3) | C1-C2 | 1.530 (3) | C1'-C2' | 1.403 (3) |
| 03-C4' | 1.357 (2) | C1-C7 | 1.547 (3) | C1'-C16 | 1.478 (3) |
| 03-C17 | 1.426 (3) | C2-C3 | 1.516 (3) | C2'-C3' | 1.370 (3) |
| 04-C3' | 1.366 (2) | C3-C4 | 1.517 (3) | C3'-C4' | 1.412 (3) |
| 04-C18 | 1.419 (3) | C4-C5 | 1.524 (3) | C4'-C5' | 1.384 (3) |
| N1-C19 | 1,466 (3) | C5-C6 | 1.545 (3) | C5'-C6' | 1.386 (3) |
| Atom-Atom-Atom | Bond angles (°) | Atom-Atom-Atom | Bond angles (°) | Atom-Atom-Atom | Bond angles (°) |
| C16-01-C3 | 117.03 (17) | 01-C3-C4 | 107.46 (19) | 04-C3'-C2' | 124.95 (18) |
| 24'-03-017 | 117 67 (18) | 03-04-05 | 113.36 (19) | 04-C3'-C4' | 115.34 (17) |
| C3'-04-C18 | 116 52 (17) | N1-C5-C4 | 107.22 (19) | C2'-C3'-C4' | 119.71 (18) |
| 19-N1-C5 | 112 2 (2) | N1-C5-C6 | 105.72 (18) | 03-C4'-C5' | 125.61 (18) |
| C19-N1-C1 | 111 92 (18) | C4-C5-C6 | 112 61 (19) | 03-C4'-C3' | 114.90 (18) |
| °5-N1-C1 | 100.67 (17) | C7-C6-C5 | 103 54 (19) | C5'-C4'-C3' | 119.00 (10) |
| N1_C1_C2 | 107.47 (19) | C6-C7-C1 | 103.37 (19) | C4'-C5'-C6' | 120 22 (10) |
| V1-01-02 V1-01-07 | 105 22 (10) | C6'-C1'-C2' | 110.70 (10) | C4-C3-C0 C1'-C6'-C5' | 120.33 (19) |
| 2 C1 C7 | 112 0 (2) | CG' C1' C16 | 122.60 (10) | | 120.4 (2) |
| 52-51-57 52-62-61 | 112.9 (2) 112.02 (10) | | 146.07 (19) | 02-010-01 | 123.1 (2) |
| L3-L2-L1 | 113.02 (19) | | 110.07 (18) | 01 616 61 | 124.3 (2) |
| 01-63-62 | 109.29 (19) | 63-62-61 | 120.62 (19) | 01-016-01 | 112.59 (18) |
| fable 4. Selected bor | nd lengths and bond angle | es of molecule 3 . | | | |
| | Bond longth (Å) | Atom-Atom | Bond length (Å) | Atom-Atom | Bond length (Å) |
| Atom-Atom | bonu length (A) | ALUIII-ALUIII | Donu icingui (A) | Atom Atom | Dona rengen (in) |
| Atom-Atom D1-C16 | 1.306 (2) | N1-H1 | 0.92 (3) | C3'-C4' | 1.478 (3) |
| Atom-Atom 01-C16 01-C3 | 1.306 (2) 1.426 (2) | N1-H1 C1-C2 | 0.92 (3) 1.606 (3) | C3'-C4' C4'-C5' | 1.478 (3) 1.320 (3) |
| Atom-Atom D1-C16 D1-C3 D2-C16 | 1.306 (2) 1.426 (2) 1.270 (3) | N1-H1 C1-C2 C2-C3 | 0.92 (3) 1.606 (3) 1.526 (3) | C3'-C4' C4'-C5' C5'-C6' | 1.478 (3) 1.320 (3) 1.358 (3) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' | 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) | N1-H1 C1-C2 C2-C3 C3-C4 | 0.92 (3) 1.606 (3) 1.526 (3) 1.467 (3) | C3'-C4' C4'-C5' C5'-C6' C19-C20 | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 | 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 | 0.92 (3) 1.606 (3) 1.526 (3) 1.467 (3) 1.584 (3) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C4' D3-C17 D4-C3' | 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 | 0.92 (3) 1.606 (3) 1.526 (3) 1.467 (3) 1.584 (3) 1.582 (3) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D3-C17 D4-C3' D4-C18 | both length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' | 0.92 (3) 1.606 (3) 1.526 (3) 1.467 (3) 1.584 (3) 1.522 (3) 1.341 (3) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C25 C20-C21 C21-C22 | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C4' D3-C17 D4-C3' D4-C18 N1-C1 | both length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.490 (3) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C2' | 0.92 (3) 1.606 (3) 1.526 (3) 1.467 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C25 C21-C22 C22-C23 | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C4' D3-C17 D4-C3' D4-C18 V1-C1 V1-C5 | Bond length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.490 (3) 1.422 (2) 1.570 (3) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C16 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C4' D3-C17 D4-C3' D4-C18 V1-C1 V1-C1 V1-C1 V1-C1 V1-C19 | Bolina length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.490 (3) 1.422 (2) 1.507 (3) 1.550 (2) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C2' C1'-C6' C1'-C16 C2'-C2' | 0.92 (3) 1.606 (3) 1.526 (3) 1.584 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.440 (3) 1.344 (3) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C22-C23 C23-C24 C24-C25 | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) |
| Atom-Atom 11-C16)1-C3)2-C16)3-C4')3-C4)3-C17)4-C3')4-C18 \t1-C1 \t1-C1 \t1-C19 \t1-C4 | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.505 (2) Bond angles (°) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom | 0.92 (3) 1.606 (3) 1.526 (3) 1.527 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.344 (3) Bond angles (°) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C38 V1-C1 V1-C5 V1-C19 Mom-Atom-Atom V16-01-C3 | Bond length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.584 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.440 (3) 1.344 (3) Bond angles (°) 112.88 (17) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom O3-C4'-C3' | 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) |
| Atom Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C18 V1-C1 V1-C5 V1-C19 Atom-Atom-Atom C16-01-C3 '4'-03-C17 | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.420 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121 10 (19) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C2' C1'-C6' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.522 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.440 (3) 1.344 (3) Bond angles (°) 112.88 (17) 113.89 (18) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom O3-C4'-C3' C4'-C5'-C6' | Bond angles (*) 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115 0 (2) |
| Atom Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C18 V1-C1 V1-C5 V1-C19 Atom-Atom-Atom C16-01-C3 C24'-03-C17 Si'-04-C18 | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.490 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.584 (3) 1.582 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.440 (3) 1.344 (3) Bond angles (°) 112.88 (17) 113.89 (18) 102.29 (17) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' | Bond angles (*) 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) |
| Atom Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C18 N1-C1 V1-C15 V1-C19 Atom-Atom-Atom C16-01-C3 C4'-03-C17 C3'-04-C18 C1-01-C3 C4'-03-C17 C3'-04-C18 C1-N1-C5 | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.490 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C6 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.443 (3) 1.344 (3) Bond angles (°) 112.88 (17) 113.89 (18) 102.29 (17) 104.55 (16) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-01 | Bond angles (*) 1.478 (3) 1.320 (3) 1.358 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) |
| Atom Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C3' D4-C18 V1-C1 V1-C5 V1-C19 Atom-Atom-Atom C16-01-C3 '24'-03-C17 '3'-04-C18 '1-03-C17 '1-03-C19 | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108 16 (16) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C4 C6-C5-C4 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.522 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.443 (3) 1.344 (3) Bond angles (°) 112.88 (17) 113.89 (18) 102.29 (17) 104.55 (16) 12110 (16) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-C1' | Bond angles (*) 1.478 (3) 1.320 (3) 1.358 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C18 V1-C1 V1-C5 V1-C19 Atom-Atom-Atom-Atom C16-01-C3 24'-03-C17 C3'-04-C18 C1-N1-C5 C1-N1-C5 S1-N1-C19 S5-N1-C19 | Bond length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C4 C6-C5-C4 C7-C6-C5 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.522 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.440 (3) 1.344 (3) Bond angles (°) 112.88 (17) 113.89 (18) 102.29 (17) 104.55 (16) 121.10 (16) 102.05 (17) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-C1' 01-C16-C1' | Bota angle (*) 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 105 46 (18) |
| Atom Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C18 V1-C1 Xiom-Atom-Atom C16-01-C3 Zi-O3-C17 23'-O4-C18 C1-N1-C19 Zi-N1-C19 Zi-N1-C19 Zi-N1-H1 | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 100.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C4 C6-C5-C4 C6-C5-C4 C7-C6-C5 C6-C7-C1 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.527 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.440 (3) 1.344 (3) 1.344 (3) 1.344 (3) 1.344 (3) 1.349 (18) 102.29 (17) 104.55 (16) 121.10 (16) 102.05 (17) 106.92 (17) | C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-C1' 01-C16-C1' C20-C19-N1 | Book Construction 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 105.46 (18) 109 10 (18) 109 10 (18) |
| Atom 11-C16 11-C3 12-C16 13-C4' 13-C17 14-C3' 14-C18 11-C5 11-C19 Atom-Atom-Atom 116-01-C3 24'-03-C17 33'-04-C18 11-N1-C5 21-N1-C19 5-N1-C19 11-N1-C5 21-N1-H1 55-N1-H1 | Bond length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.490 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C4 C6-C5-C4 C7-C6-C5 C6-C7-C1 C2'-C1'-C16 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.443 (3) Bond angles (°) 112.88 (17) 113.89 (18) 102.29 (17) 104.55 (16) 121.10 (16) 102.05 (17) 106.92 (17) 111.9 (2) | Atom C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-01 02-C16-C1' 01-C16-C1' 01-C16-C1' C20-C19-N1 C25-C20-C19 | Bond angles (°) 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 105.46 (18) 109.10 (18) 116.3 (2) |
| Atom Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C18 V1-C1 C1-01-C3 C4'-03-C17 C3'-04-C18 C1-N1-C5 C1-N1-C5 C1-N1-C19 C5-N1-C19 C1-N1-H1 C1-N1-H1 C1-N1-H1 | Bond length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) 109.4 (16) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C4 C6-C5-C4 C7-C6-C5 C6-C7-C1 C2'-C1'-C16 C6'-C1'-C16 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.584 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.443 (3) 1.344 (3) Bond angles (°) 112.88 (17) 113.89 (18) 102.29 (17) 104.55 (16) 121.10 (16) 102.05 (17) 106.92 (17) 111.9 (2) 126 77 (19) | C3'-C4' C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-01 02-C16-01 02-C16-C1' 01-C16-C1' C20-C19-N1 C25-C20-C19 C25-C20-C19 | Bond angles (*) 1.478 (3) 1.320 (3) 1.358 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 105.46 (18) 109.10 (18) 116.3 (2) 124.2 (2) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C18 V1-C1 V1-C1 V1-C1 V1-C5 V1-C19 Atom-Atom-Atom C16-01-C3 24'-03-C17 C3'-04-C18 C1-N1-C5 C1-N1-C19 C5-N1-C19 C1-N1-C19 C5-N1-C19 C1-N1-H1 C1-N1-H1 C1-N1-H1 V1-C17 | Bond length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) 109.4 (16) 115.4 (15) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C4 C6-C5-C4 C7-C6-C5 C6-C7-C1 C2'-C1'-C16 C6'-C1'-C16 C1'-C1/6 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.522 (3) 1.522 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.440 (3) Bond angles (°) 112.88 (17) 112.88 (17) 123.88 (17) 124.88 (1 | C3'-C4' C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C23 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-C1' 02-C16-C1' 01-C16-C1' C20-C19-N1 C25-C20-C19 C25-C20-C21 C19-C20-C21 | Book (1) 1.478 (3) 1.320 (3) 1.358 (3) 1.391 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 105.46 (18) 109.10 (18) 116.3 (2) 124.2 (2) 110.4 (2) 114.4 (2) |
| Atom Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C18 V1-C1 V1-C1 V1-C1 V1-C19 Atom-Atom-Atom C16-01-C3 24'-03-C17 23'-04-C18 C1-N1-C5 C1-N1-C5 21-N1-C19 25-N1-C19 Z1-N1-H1 55-N1-H1 C19-N1-H1 V1-C1-C7 V1-C1-C7 V1-C1-C7 V1-C1-C7 | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) 109.4 (16) 115.4 (15) 99.06 (16) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C2' C1'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C4 C6-C7-C1 C2'-C1'-C16 C6'-C7-C1 C2'-C1'-C16 C6'-C1'-C16 C1'-C2'-C3' | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.527 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.440 (3) 1.344 (3) 1.344 (3) 1.344 (3) 1.344 (3) 1.348 (17) 112.88 (17) 112.88 (17) 113.89 (18) 102.29 (17) 104.55 (16) 121.10 (16) 102.05 (17) 106.92 (17) 106.92 (17) 111.9 (2) 126.77 (18) 114.6 (2) 110 (2) | CG3'-C4' C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-01 02-C16-01 02-C16-C1' 01-C16-C1' C20-C19-N1 C25-C20-C21 C19-C20-C21 C19-C20-C21 C19-C20-C21 C30 | Book Construction 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 105.46 (18) 109.10 (18) 116.3 (2) 124.2 (2) 119.4 (2) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C3' D4-C18 V1-C1 V1-C5 V1-C19 Atom-Atom-Atom C16-01-C3 C4'-03-C17 D3'-04-C18 C1-N1-C5 C1-N1-C19 C5-N1-C19 C5-N1-C19 C5-N1-C19 C5-N1-C19 C1-N1 | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.490 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) 109.4 (16) 115.4 (15) 99.06 (16) 105.04 (17) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C6 N1-C5-C4 C6-C5-C4 C7-C6-C5 C6-C7-C1 C2'-C1'-C16 C6'-C1'-C16 C6'-C1'-C16 C1'-C2'-C3' O4-C3'-C2' O4-C3'-C2' | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.443 (3) 1.344 (3) Bond angles (°) 112.88 (17) 113.89 (18) 102.29 (17) 104.55 (16) 121.10 (16) 102.05 (17) 106.92 (17) 106.92 (17) 111.9 (2) 126.77 (18) 114.6 (2) 119.9 (2) 114.6 (2) 119.9 (2) | CG3'-C4' C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-C1 02-C16-C1' 01-C16-C1' 01-C16-C1' C25-C20-C21 C19-C20-C21 C19-C20-C21 C19-C20-C21 C22-C21-C20 C22-C21-C20 | Bond angles (°) 1.478 (3) 1.320 (3) 1.358 (3) 1.396 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 105.46 (18) 109.10 (18) 116.3 (2) 124.2 (2) 119.4 (2) 118.6 (3) 116.4 (3) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C3' D4-C18 N1-C1 V1-C5 V1-C1 V1-C5 V1-C19 Atom-Atom-Atom C16-01-C3 C4'-03-C17 C3'-04-C18 C1-N1-C5 C1-N1-C5 C1-N1-C5 C1-N1-C5 C1-N1-C19 C5-N1-C19 C5-N1-C19 C1-N1-H1 C5-N1-H1 V1-C1-C7 V1-C1-C2 C7-C1-C2 C7-C1-C2 C7-C1-C2 C1-C2 C7-C1-C2 C1-C1 C1-C3 C1-C1 C1-C2 C7-C1-C2 C1-C1 C1-C2 C1-C1 C1-C2 C1-C2 C1-C1 C1-C2 C1-C2 C1-C2 C1-C2 C1-C2 C1-C3 C1-C3 C1-C2 C1-C2 C1-C2 C1-C2 C1-C2 C1-C2 C1-C2 C1-C2 C1-C2 C1-C2 C1-C3 C1-C2 C1-C2 C1-C2 C1-C2 C1-C2 C1-C3 C1-C3 C1-C2 C | Bond rengin (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) 109.4 (16) 115.4 (15) 99.06 (16) 105.04 (17) 119.44 (17) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4+C5 N1-C5-C6 N1-C5-C6 N1-C5-C4 C6-C7-C1 C2'-C3' O4-C3'-C1'-C16 C1'-C2'-C3' O4-C3'-C2' O4-C3'-C2' O4-C3'-C2' O4-C3'-C2' O4-C3'-C2' O4-C3'-C2' | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.522 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.443 (3) 1.344 (3) Bond angles (°) 112.88 (17) 113.89 (18) 102.29 (17) 104.55 (16) 121.10 (16) 102.05 (17) 106.92 (17) 106.92 (17) 111.9 (2) 126.77 (18) 114.6 (2) 119.9 (2) 116.3 (2) 120.55 (16) | C3'-C4' C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-01 02-C16-01 02-C16-01 02-C16-C1' C10-C16-C1' C20-C19-N1 C25-C20-C19 C25-C20-C21 C19-C20-C21 C22-C21-C20 C21-C22-C23 C22 | Bond angles (*) 1.478 (3) 1.320 (3) 1.358 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 109.10 (18) 116.3 (2) 124.2 (2) 119.4 (2) 118.6 (3) 116.1 (3) |
| Atom-Atom D1-C16 D1-C3 D2-C16 D3-C4' D3-C17 D4-C3' D4-C18 N1-C5 N1-C1 N1-C5 N1-C19 Atom-Atom-Atom- C16-01-C3 C4'-03-C17 C3'-04-C18 C1-N1-C5 C1-N1-C19 C1-N1-C1 | bold length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.490 (3) 1.422 (2) 1.507 (3) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 1121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) 109.4 (16) 115.4 (15) 99.06 (16) 105.04 (17) 119.44 (17) 114.75 (17) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C6' C1'-C16 C2'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C4 C6-C5-C4 C7-C6-C5 C6-C7-C1 C2'-C1'-C16 C1'-C2'-C3' O4-C3'-C2' O4-C3'-C2' O4-C3'-C4' C2'-C3'-C4' C2'-C3'-C4' | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.522 (3) 1.522 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.443 (3) 1.344 (3) Bond angles (°) 112.88 (17) 112.88 (17) 112.88 (17) 112.88 (17) 112.88 (17) 112.88 (17) 112.88 (17) 112.88 (17) 104.55 (16) 121.10 (16) 102.05 (17) 106.92 (17) 111.9 (2) 126.77 (18) 114.6 (2) 119.9 (2) 116.3 (2) 123.85 (18) | C3'-C4' C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C20-C21 C21-C22 C22-C23 C23-C24 C24-C23 C4'-C3' C4'-C3'-C6' C5'-C6'-C1' 02-C16-C1' 02-C16-C1' 01-C16-C1' C20-C19-N1 C25-C20-C19 C25-C20-C19 C25-C20-C21 C19-C20-C21 C22-C21-C20 C21-C22-C23 C22-C23-C24 C22-C23 C22-C23-C24 C22-C23 C22-C2 | Bond angles (*) 1.478 (3) 1.320 (3) 1.358 (3) 1.391 (3) 1.422 (4) 1.300 (4) 1.375 (5) 1.428 (5) 1.277 (4) Bond angles (°) 119.99 (19) 115.0 (2) 124.22 (19) 126.0 (2) 128.48 (18) 105.46 (18) 109.10 (18) 116.3 (2) 124.2 (2) 119.4 (2) 118.6 (3) 116.1 (3) 125.7 (3) |
| Atom Atom O1-C16 O1-C3 O2-C16 O3-C4' O3-C17 O4-C18 V1-C1 V1-C1 V1-C1 V1-C19 Atom-Atom-Atom T16-01-C3 24'-03-C17 C3'-04-C18 C1-N1-C5 C1-N1-C19 Z5-N1-C19 Z1-N1-C19 Z1-N1-C19 Z1-N1-H1 V1-C1-C2 Z'N-C1-H1 X1-C1-C2 X1-C1-C2 X1-C1-C2 X1-C1-C2 X1-C1-C2 X1-C1-C2 X1-C1-C2 X1-C2-C1 X1-C2-C1 | Bond length (A) 1.306 (2) 1.426 (2) 1.270 (3) 1.322 (3) 1.511 (3) 1.295 (2) 1.400 (3) 1.422 (2) 1.507 (3) 1.550 (2) Bond angles (°) 110.32 (17) 121.10 (19) 118.22 (18) 103.74 (15) 108.16 (16) 112.08 (15) 107.2 (15) 109.4 (16) 115.4 (15) 99.06 (16) 105.04 (17) 114.75 (17) 111.96 (18) | N1-H1 C1-C2 C2-C3 C3-C4 C1-C7 C6-C7 C1'-C2' C1'-C2' C1'-C3' Atom-Atom-Atom C4-C3-C2 C3-C4-C5 N1-C5-C6 N1-C5-C6 N1-C5-C4 C6-C7-C1 C2'-C1'-C16 C6'-C1'-C16 C1'-C2'-C3' O4-C3'-C2' O4-C3'-C4' C2'-C3'-C4' C5'-C4-C3 | 0.92 (3) 1.606 (3) 1.526 (3) 1.526 (3) 1.527 (3) 1.584 (3) 1.522 (3) 1.341 (3) 1.443 (3) 1.443 (3) 1.440 (3) 1.344 (3) 1.344 (3) 1.344 (3) 1.348 (17) 112.88 (17) 112.88 (17) 112.88 (17) 112.88 (17) 112.85 (16) 121.10 (16) 102.05 (17) 106.92 (17) 111.9 (2) 126.77 (18) 114.6 (2) 119.9 (2) 116.3 (2) 123.85 (18) 119.1 (2) | CG3'-C4' C3'-C4' C4'-C5' C5'-C6' C19-C20 C20-C25 C22-C23 C23-C24 C24-C25 Atom-Atom-Atom 03-C4'-C3' C4'-C5'-C6' C5'-C6'-C1' 02-C16-C1' 02-C16-C1' 01-C16-C1' C20-C19-N1 C25-C20-C21 C19-C20-C21 C22-C23-C24 C22-C23-C24 C22-C23-C24 C25-C24-C23 C22-C23-C24 C25-C24-C23 | Book Book <th< td=""></th<> |

| D-HA | d(D-H), Å | d(HA), Å | d(DA), Å | ∠(DHA), ° | Symmetry |
|-------------|-----------|----------|----------|-----------|---|
| Compound 1 | | | | | |
| C17-H17a02 | 0.96 | 2.47 | 3.405(9) | 164 | 1+ <i>x</i> , <i>y</i> , <i>z</i> |
| Compound 2 | | | | | |
| C18-H18c02 | 0.96 | 2.53 | 3.268(3) | 134 | 1- <i>x</i> , 1- <i>y</i> , 2- <i>z</i> |
| Compound 3 | | | | | |
| N1-HCl1 | 0.93(3) | 2.42(3) | 3.337(2) | 174(2) | - |
| C2-H2aCl1 | 0.97 | 2.76 | 3.499(2) | 133 | - |
| C4-H4bCl1 | 0.97 | 2.82 | 3.552(3) | 133 | - |
| C7-H7a04 | 0.97 | 2.20 | 3.106(3) | 155 | -X, -Y, -Z |
| C19-H19bCl1 | 0.97 | 2.64 | 3.577(2) | 162 | 1-x, -1-y, -z |

Bond length in the molecules **1-3** closely comparable with only small variations around the N1 nitrogen atom. N1-C1 (1.466(10) Å), N1-C5 (1.468(10) Å) bonds in molecule **1** and N1-C1 (1.466(10) Å), N1-C5 (1.468(10) Å) and N1-C19 1.466(3) Å bonds in molecule **2** are almost the same. In case molecule **3** with protonation of N1 atom those bond lengths equals to 1.422(2), 1.507(3), 1.550(2) Å, respectively (Tables 2-4).

In crystal structures **1** and **2**, the molecules are located in the distance of van der Waals interactions. However, the crystal structure indicates that the molecules in the crystals are associated by intermolecular weak C-H...O interactions (Table 5). Crystal **3** is the hydrochloride salt. The H-bond

between the anion Cl and the proton of the N atom is observed in the crystal, the H-bond parameters are the following: Cl1…N1 3.337(2) Å, Cl1…H 2.42(3) Å, Cl1…HN1 174(2)°. A weak H-bond of C7-H…O4 type is observed, and also π - π interactions between the aromatic sites of the veratroyloxy group of molecules transformed by the center of symmetry.

4. Conclusion

Crystal structures of convolvine alkaloid and its two derivatives were deduced by single crystal XRD analysis. Piperidine rings in compounds adopt chair conformation. Hydrogen atom and methyl- and benzyl-substituents located in nitrogen atom of **1-3** occupy equatorial positions. The veratroyloxy group containing in all compound molecules is an α -axial oriented relative to the tropane core. Free rotation of dimethoxyphenyl- group around the C16-C1' bond leads different rotamers in crystals of compound **1-3**.

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Supporting information S

CCDC 1935317 (1), CCDC 1935318 (2) and CCDC 1935324 (3) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/, or by e-mailing data request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

Disclosure statement 💿

Conflict of interests: The authors declare that they have no conflict of interest.

Author contributions: All authors contributed equally to this work.

Ethical approval: All ethical guidelines have been adhered.

Sample availability: Samples of the compounds are available from the author.

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