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Synthesis, structural chemistry and antimicrobial activity of -(-) borneol derivative

Research Article

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Abstract: Borneol is a monoterpene that is a part of traditional Chinese and Japanese medicine. (-) borneol reacted with methanesulfonyl chloride in THF/pyridine to afford the new 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate derivative in excellent yield. The product is characterized by H1NMR, C^{13}NMR, mass spectroscopy as well as elemental analysis and its structure was identified by X-ray single crystal diffraction. The packing of 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methanesulfonate exhibits the non-classical C—H···O hydrogen bonding in C(4) and $R^2_2(8)$ chain and ring motifs as structural determinants. This was also confirmed by the analysis of Hirshfeld surfaces. The 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate antimicrobial activity was tested and compared with its parent (-) borneol against three different pathogens. Particularly, 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate showed high sensitivity, compared to Chloramphenicol reference material, against *Escherichia coli*.

Keywords: *(-)borneol • X-ray single crystal • Hirshfeld surfaces • C-H···O weak hydrogen bonding • Antimicrobial activity*

1. Introduction

Most of the natural products are known to show unique medicinal or biological properties, and normally they exhibit tolerance with healthy tissues. The interest in the biological activities of plant extracts had been the subject of intense scientific investigation [1]. Among these are terpenes where camphor and borneol are common monoterpenes. In the nineteenth century, borneol was known to be prepared by the reduction of d- or l-camphor by means of sodium in alcoholic solution to produce a mixture of borneol and isoborneol [2], the latter which is stereoisomeric with the former, recently showed dual viricidal activity against human herpes simplex virus-1 (HSV-1) [3]. Since ancient civilizations [4], the two optically active forms of borneol {d- and l- or +(-) and -(-)}, derived from pine oil and cedar leaf oil as well as ginger oil, were used as disinfectants [5], deodorants [6], food and cosmetics additives [7] with well known analgesic, anti-inflammatory and antibacterial enhancing effects [8]. Borneol worldwide consumption is in the range of 1-10 metric tonnes per year [9] as it consumed excessively in southeast Asian countries, particularly in combined formulas for preventing cardiovascular diseases [10].

In addition, (-)- borneol was found to have a highly efficacious positive modulating action at mammalian GABA (γ-Aminobutyric acid) inhibitory neuro-transmission receptors, as did its enantiopure (+)- borneol [11]. Moreover, a mixture of Bismuth subgallate and borneol was found to show high antitumor effect against melanoma skin cancer [8a,12], and the bornyl (3,4,5-trihydroxy)-cinnamate derivative is showing an optimized human neutrophil elastase (HNE) inhibitory effect [13]. The great affinity of borneol with water solvates inspired its use as a part of supramolecular intercalation host-guest molecules. Weber et al effectively used borneol as a part of macrocyclic host molecule by attaching with ethynylidene or butadiynilidene central units and studied the inclusion behavior of many types of guest molecules that all found to depend on the borneol host

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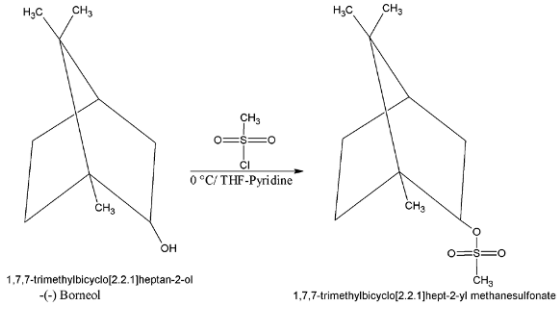
supramolecular hydrogen bonding interactions with the guests [14]. Recently, the solid state syntheses were also implemented in the preparation of borneol-methyl-cyclodextrin, a highly water-soluble inclusion complex by both supercritical carbon dioxide processing and sealed heating treatment at moderate temperature and pressure [15].

With the given background on the richness of borneol chemistry, we present here the synthesis, characterization, structural analysis and antimicrobial activity of (1S-endo)-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate derivative. The compound was prepared as a part of our ongoing research in the syntheses of new chiral organic ligands [16], preferably water soluble, in order to study the capacity of these derivatives for transferring their stereochemical character to transition metals, and subsequently to specified catalytic sites. An example on that is the reaction of monocyclopentadienyl titanium dichlorides with (1S-endo)-(-) borneol that exposed a chiral organometallic complex [17].

2. Experimental Procedure

2.1 Syntheses

5% excess of methanesulfonyl chloride (4g, 0.036 mol) was dissolved in 10 mL dry THF then added slowly to a stirred solution of(1S-endo)-(-)borneol (5.4 g, 0.035 mol) in 20 mL dry THF. After the reaction mixture was stirred approximately for 30 min at 0°C, 10 mL of absolute pyridine was added. The reaction was sealed then stirred for 24 h at room temperature. Pyridinium chloride precipitate was filtered off. The volume of the solution was concentrated under reduced pressure then transferred to a separating funnel and washed with 40 mL dichloromethane and 100 mL of distilled H_2O. By extracting the organic layer and evaporating the solvent in vacuo, pure white powder of 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate was collected in 95% yield. ^1H NMR ($CDCl_3$): δ (ppm) 0.86 (br, 6H, $2CH_3$), 1.22. (m, br, 3H, $CHCH_3$), 1.67 (com, 4H, CH_2CH_2), 2.34 (m, 2H, $OCHCH_2CH$), 2.50 (m, 1H, CH) 3.15 (s, 3H, SCH_3) 4.71 (2m, 1H, OCH). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ (ppm) 13.40 [s-C- singlet, CCH_3], 19.11, 20.10 [2 s-C- singlet, $(CH_3)_2C$], 26.76 [s-C- singlet, CH_3CCH_3], 28.01 [s-C- singlet, $CHCH_2CH_2C$], 36.40 [s-C- singlet, $CHCH_2CH_2C$], 36.85 [s-C- singlet, $OCHCH_2CH$], 44.97 [s-C- singlet, SCH_3], 47.85 [s-C- singlet, $OCHCH_2CH$], 49.67 [s-C- singlet, CH_3C], 87.34 [s-C- singlet, OCH]. EI-Ms = 232.1 M^+, Elem. Anal. calc. C, 56.86; H, 8.68; S, 13.80%. found: C, 56.41; H, 8.85; S, 13.93, Crystals suitable for X-ray study were grown by slow diffusion of n-hexane-diethyl ether (1:1 v/v) into a dichloromethane solution of 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate.



1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol -(-) Borneol

1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methanesulfonate

Scheme 1. Structural formula and synthetic conditions of 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate

2.2. Experimental

Elemental analyses were carried out on Elemental Vario EL analyzer. High-resolution ^1H and $^{13}C\{^1H\}$ NMR spectra were recorded on a Bruker DRX 250 spectrometer at 298 K with frequencies as follows: ^1H 250.12 MHz, $^{13}C\{^1H\}$ 62.9 MHz. Chemical shifts in the ^1H and $^{13}C\{^1H\}$ NMR spectra were measured relative to the partially deuterated solvent peaks, which are reported relative to TMS. EI-MS were recorded on Finnigan 711A (8 kV) and reported as mass/charge (m/z).

Suitable colorless block single crystal of 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate was selected under optical microscope, glued and mounted onto thin glass capillary. Diffraction data were collected using a Rigaku Raxis RAPID diffractometer equipped with imaging plate area detector utilizing Mo-K_α radiation (λ = 0.71073 Å) with graphite monochromator. The data were collected at a temperature of 298±2 K to a maximum 2θ of 55.0° with crystal-to-detector distance was 127.40 mm. A total of 108 oscillation images in three sweeps were collected using ω-scans from 20.0° to 200.0° at χ=0.0°, ψ=0.0° and at χ=54.0°, ψ=120.0° and at χ=54.0°, ψ=240.0°. Preliminary orientation matrices, unit cell determination and data reduction and integration were performed using Crystal Clear package [18]. The data were empirically corrected against absorption and final cell parameters were obtained by refinement of the positions of reflections with $I > 10\sigma_I$ after integration using Crystal Clear package [18]. The structure was solved by direct methods and refined by full-matrix least squares on all $|F^2|$ data using SHELXTL package [19]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using their appropriate riding model with isotropic atomic displacement factor $U_{iso}(H)$ 1.2 times U_{eq} of the pivot atom. Figs. 1 and 2 were created using the DIAMOND package [20]. Crystal data and refinement details: Empirical formula: $C_{11}H_{20}O_3S$,

Formula weight: 232.34, Crystal system: Orthorhombic, space group: $P2_12_12_1$, Unit cell dimensions: a = 8.2788(17) Å, b = 9.3925(19) Å, c = 15.857(3) Å, V: 1233.0(4) A^3, Z: 4, Calculated density: 1.252 $Mg\,m^{-3}$, Crystal size: 0.4×0.25×0.25 mm, θ-range for data collection: 3.3-27.5°, Limiting indices: -10<=h<=10, -12<=k<=12, -20<=l<=20, Reflections collected/ unique: 29807 / 2824, R_{int}: 0.034, Completeness to θ= 25.00°: 99.8%, Data/restraints/parameters: 2824/0/140, Goodness-of-fit on F^2: 1.03, Final R indices [I>2σ(I)]: R1 = 0.032, wR2 = 0.094, Largest diff. Peak/hole : 0.23/-0.20 e A^{-3}. CIF file containing complete information on the studied structure was deposited with CCDC, deposition number 759725, and is freely available upon request from the following web site: www.ccdc.cam.ac.uk/data_request/cif. Hirshfeld surfaces were calculated with CrystalExplorer package [21].

2.3. Antimicrobial activity

The disc diffusion method was employed for the determination of antimicrobial activity using agar nutrient as the medium [22]. Staphylococcus aureus ATCC6538, Escherichia coli ATCC25292 and Candida albicans ATCC27853 strains were used as microbial pathogens. 10^{-3} M Stock solutions were prepared by dissolving (1S-endo)-(-)borneol and 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate in DCM. In a typical procedure, a well was made on the agar medium inoculated with microorganisms. The well was filled with the test solution using a micropipette and the plate was incubated at 410 K for 24 h after being kept in the refrigerator for 2 h. During this period, the test solution is diffused and the growth of the inoculated microorganism is examined. The diameters of the inhibition zones were measured in millimeters.

3. Results and Discussion

The trimethylbicyclo-hept-2-yl methane sulfonate is crystallized in the $P2_12_12_1$ Orthorhombic acentric space group with Flack parameter of 0.04(7) and the chirality of C3 (S-endo), C7 (R-endo) and C8 (S-endo) resemble those of the parent (1S-endo)-(-) borneol. The interatomic bond distances and angles summarized in Table 1 show no unusual values and the thermal ellipsoidal drawing is presented in Fig. 1.

The methane sulfonate group adopts a distorted tetrahedral geometry around the central sulfur atom with two double and two single bonds. The least-square mean best-fit planes of C3-C6-C7-C8 and C2-C3-C4-C8 are intersecting with angle of 68.48°and the plane of C1-C3-C5-C8 defining an interplanar angle with those at 56.79° and 54.76°, respectively. The enhanced solubility of the new derivative in aqueous solutions, compared to borneol, was attracting our attention towards supramolecular interactions of molecules with water solvates. One can expect that (1S-endo)-(-) borneol hydrogen bonding motif is mainly a one dimensional chain nature with absence of aromaticity and presence of one terminal hydroxyl group free contact. From a supramolecular chemistry perspective, the methane sulphonate, with two acceptors oxygen atoms and one donor methane carbon atom, is providing structural motifs of the C—H···O type so that a hydrogen bonding network structure can be expected. It is known that the use of weak hydrogen bonding like C—H···O in crystal engineering is a hurdle as it is marginally electrostatic with weak covalency [23]. Although it's ubiquitous existence in biological structures [24], the C—H···O hydrogen bonding, in most known cases, is of supportive nature in crystal packing [25].

Table 1 reports the hydrogen bonding D—H···A geometries of the trimethylbicyclo-hept-2-yl methane sulfonate and Fig. 2 depicts a part of the unit cell packing with hydrogen bonding interactions shown. Two non-classical C—H···O hydrogen bonding interactions are interconnecting the molecules in the ac-plane, forming a 2-dimensional brick-like network. The interaction patterns were identified as C(4) first-level chain and $R^2_2(8)$ second-level ring motifs, as earlier described [26].

The non-classical hydrogen bonding, in particular C—H···O was a part of scientific disputes in the last decade [23]. Cotton and co-workers stated that the weak

Table 1. Selected bond distances [Å] and interatomic angles [°].

S1—O2	1.4203 (13) Å	C3-C6-H6A	111.3°
S1—O1	1.4311 (12) Å	O1-S1-O3	104.26 (7)°
C5—C11	1.535 (2) Å	C7-C6-H6A	111.3°
S1—O3	1.5659 (11) Å	O2-S1-C10	109.19 (9)°
C5—C9	1.539 (2) Å	O1-S1-C10	108.75 (9)°
S1—C10	1.7479 (18) Å	O3-S1-C10	103.89 (8)°
C5—C8	1.5614 (19) Å	C7-O3-S1	119.52 (8)°
C1—C8	1.509 (2) Å	S1-C10-H10A	109.5°
C6—C7	1.5403 (19) Å	C8-C7-C6	104.96 (11)°
C2—C3	1.526 (3) Å	O3-C7-H7	109.9°
O3—C7	1.4754 (16) Å	C2-C3-C6	106.86 (14)°
C3—C6	1.539 (2) Å	C1-C8-C4	114.30 (14)°
		C6-C3-C5	102.82 (12)°
		C3-C2-C4	102.82 (12)°
O2-S1-O1	119.07 (8)°	C1-C8-C7	114.41 (13)°
C3-C6-C7	102.11 (11)°	C7-C8-C4	107.09 (12)°
O2-S1-O3	110.57 (7)°		

Hydrogen bonding geometries (Å and °)

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10B···O2^{i}	0.96	2.55	3.476 (2)	163
C10—H10C···O1^{ii}	0.96	2.56	3.504 (2)	167

Symmetry codes: (i) x+1/2, −y+3/2, −z+1; (ii) x−1/2, −y+3/2, -z+1.

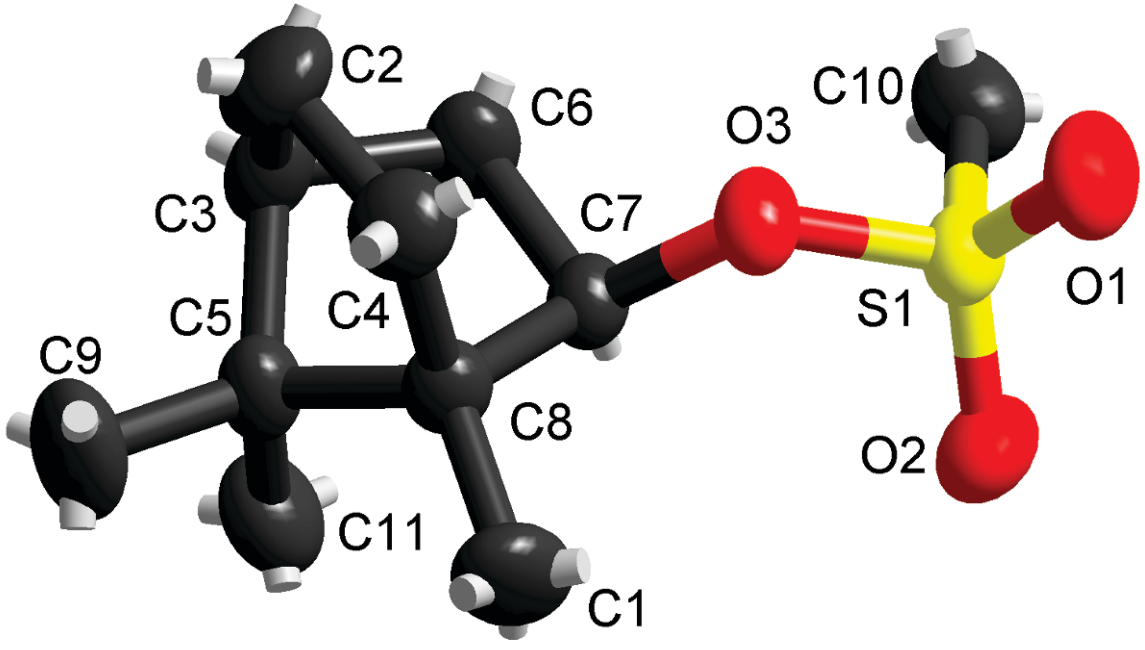


Figure 1. Atomic numbering scheme with atomic displacement ellipsoids shown at 50% probability level. Hydrogen atoms were omitted for clarity.

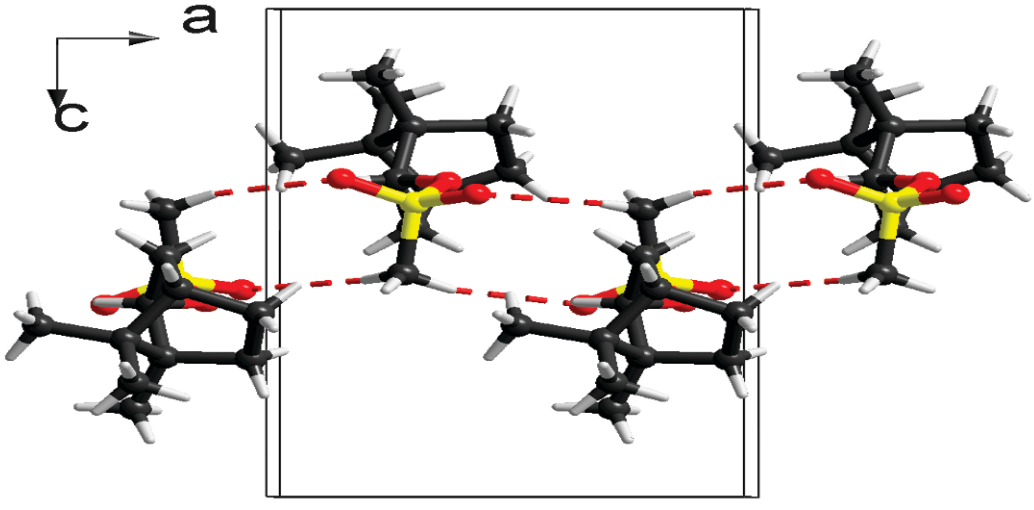


Figure 2. *ac*-plane perspective drawing showing the intermolecular C—H•••O hydrogen bonding motifs presented as dotted lines.

non-conventional C–H···O hydrogen bond represents nothing more than a classical van der Waals interaction [27]. That was later falsified by the seminal work of Desiraju and Steiner [28], as their statistical analyses of Cambridge Structural Database (CSD) hits comprising C—H···O fragments showed a gradual decrease in directionality for C–H···O interactions with decreasing C–H polarization from vinyl donors to methyl groups and is still dissimilar with the isotropic behaviour for H···H contacts [28]. Moreover, the geometrical parameters of the conventional O—H···O hydrogen bonds are confined within a narrow distance–angle cluster (shorter H···A distance at linear D—H···A angle) due to its highly inherited covalency character, but this is definitely not the case for the weak C—H···O bonds with an inverse distance–angle correlation (long H···A distance at small D—H···A angle) that is characteristic of any electrostatic interaction like hydrogen bonding [28]. Desiraju then stated 'A C–H···O soft hydrogen bond does not become a van der Waals contact just because the H···O distance crosses an arbitrary threshold'. Meanwhile, not every C–H···O contact is a hydrogen bond. it was

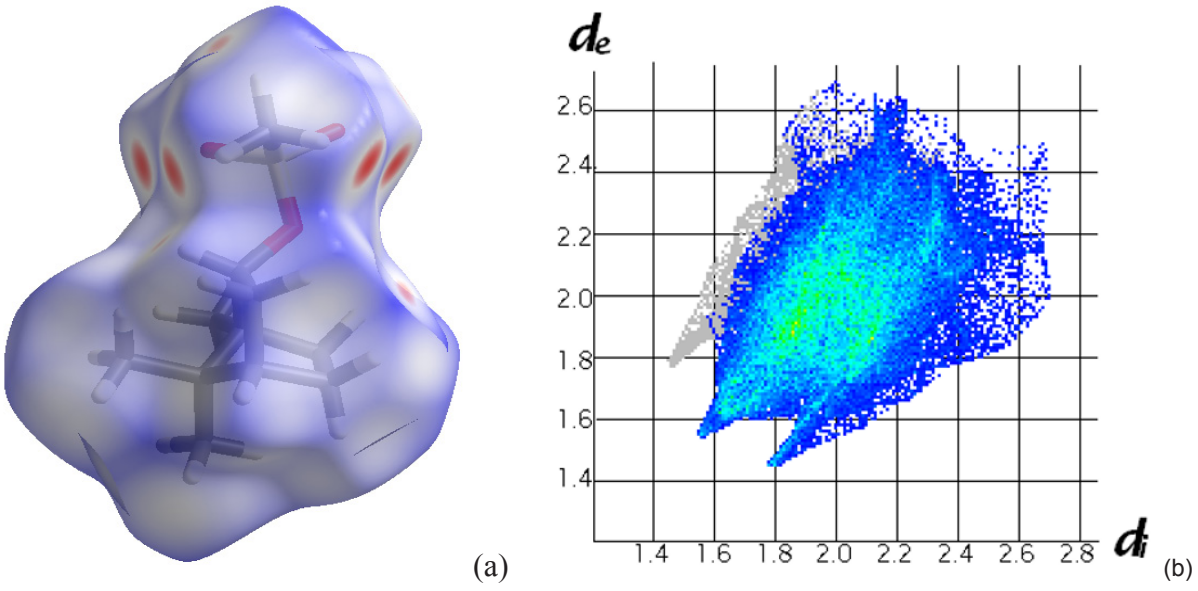


Figure 3. (a) Hirshfeld surface of trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate mapped with normalized electonic density. (b) 2D-Fingerprint plots for trimethylbicyclo hept-2-yl methane sulfonate resolved into O…H contacts. The full fingerprint appears beneath in grey shadow.

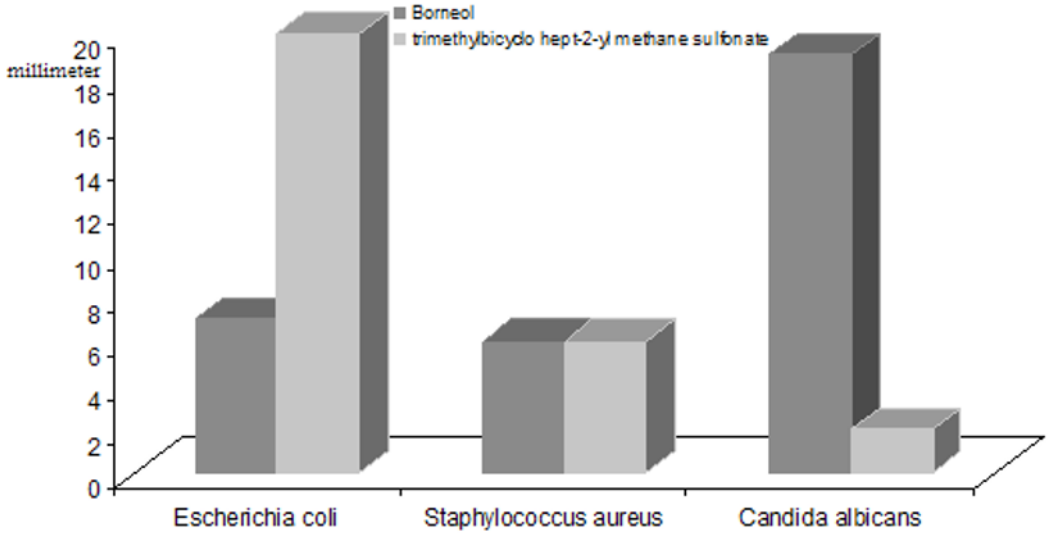


Figure 4. Plot for inhibition zones in millimeter for (-)borneol and trimethylbicyclo[2.2.1]hept-2-yl methane sulfonate against three selected and isolated pathogens.

shown that some C–H···O contacts have non-negative interaction energies [29]. What is undisputed is that there are certain types of hydrogen bonds, formed by C–H groups activated by a neighbor heteroatom, that are actually indistinguishable from classical hydrogen bonds [28,30].

In order to quantify the effect of C–H···O interactions in guiding the packing preferences of trimethylbicyclo hept-2-yl methane sulfonate, we aimed to study its procrystal electronic density. The Hirshfled surfaces, based on the Hirshfeld stockholder partitioning scheme [31], is a method to divide electron densities into fragment contributions with well-defined molecular surfaces. The Hirshfeld surface is a crystal property with a whole-of-molecule approach and is defined by a relation with the sum of spherical atoms electronic densities from the promolecule divided by the same sum for the procrystal [32]. Hirshfeld surfaces partitioning any scalar density into atomic then molecular contributions with respect to a reference value of the scalar in the isolated atomic

density derived from Clementi-Roetti's near Hartree–Fock atomic wave functions [33]. Fig. 3a depicts the d_{norm} Hirshfeld curvatures for trimethylbicyclo[2.2.1] hept-2-yl methane sulfonate and is displayed using a blue-red–white color scheme, where red highlights shorter contacts, white for contacts around the vdW separation.

Intense and large red spots, as internuclear separations decrease, are observed at the two methane sulfonate oxygen atoms and the methyl two hydrogens. Pale and small spot is also recognized at the cyclic part that specify other contact rather than C–H···O. In Fig. 3b, a two-dimensional fingerprint plot of intermolecular interactions in the crystal with d_e are distances to the nearest atoms outside the molecule and inside, di, are defined. O···H and H···O intermolecular contacts are only highlighted and the portions of the surface where hydrogen is the closest atom inside the surface, and oxygen is the nearest atom outside the surface, and vice versa, are coloured. The area of these highlighted surface patches comprises more than 85% of the total Hirshfeld surface area for this molecule. The rest of 14.6% could be identified as H···H isotropic non-directional van der Waals interactions (*i.e.*, C–H···H-C).

The *in-vitro* antimicrobial activity of trimethylbicyclo-hept-2-yl methane sulfonate and (-)borneol have been screened against *Staphylococus aureus*, *Candida Albicans* and *Escherichia coli* pathogenic strains. As the results presented in Fig. 4, the trimethylbicyclo-hept-2-yl methane sulfonate has high activity against *Escherichia coli* with inhibition zones are 6, 2 and 20 mm for *Staphylococus aureus*, *Candida Albicans* and *Escherichia coli* respectively. The (-)borneol showed high activity against *Candida Albicans* with inhibition zones are 6, 19 and 7 mm for *Staphylococus aureus*, *Candida Albicans* and *Escherichia coli*, respectively. The antibacterial effect of trimethylbicyclo-hept-2-yl methane sulfonate is comparable with that of the reference substance chloramphenicol (22 mm for *Escherichia coli*).

4. Conclusions

We presented a new derivative of the chemically and medicinally rich (-)borneol monoterpene. The presented structure of the trimethylbicyclo-hept-2-yl methane sulfonate is a rare unique example on the role of non-classical C—H···O hydrogen bonding as a solely structural determinant. That was also approved by plotting its Hirshfeld surfaces and analysis of its whole-of-molecule intermolecular interactions. Synergetic antibacterial-antifungal effect is expected with a 50:50 mixture of (-)borneol and the new trimethylbicyclo hept-2-yl methane sulfonate derivative.

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