STRUCTURE ANALYSIS OF NEWLY SYNTHESIZED STEROIDAL TETRAZOLES

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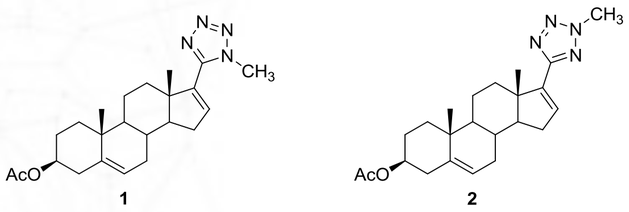
Introduction

Bearing in mind that androstane derivatives with tethered heterocyclic groups at position C–17 show anticancer activity, the aim of this study was a synthesis of novel steroidal derivatives with a tetrazole ring as a substituent at C–17 position, as well as a detailed investigation of their structures. The crystal structures of the two novel androstane derivatives: 3β-acetoxy-17-(1-methyl-1*H*-tetrazol-5-yl)androsta-5,16-diene (**1**) и 3β-acetoxy-17-(2-methyl-2*H*-tetrazol-5-yl)androsta-5,16-diene (**2**) were determined at room temperature.

Methods

COMPOUNDS

1. 3β-acetoxy-17-(1-methyl-1*H*-tetrazol-5-yl)androsta-5,16-diene (**1**)
2. 3β-acetoxy-17-(2-methyl-2*H*-tetrazol-5-yl)androsta-5,16-diene (**2**)



X-RAY CRYSTALLOGRAPHY

The diffraction data for **1** (MoKα, λ = 0.71071 Å) and **2** (CuKα, λ = 1.54184 Å) were collected at room temperature on Rigaku (Oxford Diffraction) Gemini S diffractometer using program package CrysAlis CCD. The data reduction was performed with program package CrysAlis RED. Collected data were corrected for absorption effects by using Multi-scan absorption correction. The structures were solved by direct methods using SHELXT. The structures were refined by full-matrix least-squares procedures on F^2 using SHELXL-2018/3 program.

Crystallographic data for $C_{23}H_{32}N_4O_2$ (1)	
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell parameters	a = 9.7390 (5) Å b = 7.3412 (3) Å c = 30.5185 (15) Å β = 96.970 (5)°
Data/Restrains/Parameters	7761/1/547
Goodness-of-fit on F^2	1.036
Final R indices [$I > 2\sigma(I)$]	$R1$ = 0.0591 $wR2$ = 0.1148
R indices [all data]	$R1$ = 0.1157 $wR2$ = 0.1387

Crystallographic data for $C_{23}H_{32}N_4O_2$ (2)	
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Unit cell parameters	a = 7.3721(2) Å b = 9.8807(2) Å c = 30.1493(9) Å
Data/Restrains/Parameters	4186/0/267
Goodness-of-fit on F^2	1.034
Final R indices [$I > 2\sigma(I)$]	$R1$ = 0.0411 $wR2$ = 0.1084
R indices [all data]	$R1$ = 0.0519 $wR2$ = 0.1183

HIRSHFELD SURFACE ANALYSIS AND 2D FINGERPRINT PLOTS

CrystalExplorer 17.5 was used to analyze Hirshfeld surface and associated two-dimensional fingerprint plots for compounds **1** and **2**.

Hirshfeld surface analysis and 2D fingerprint plots

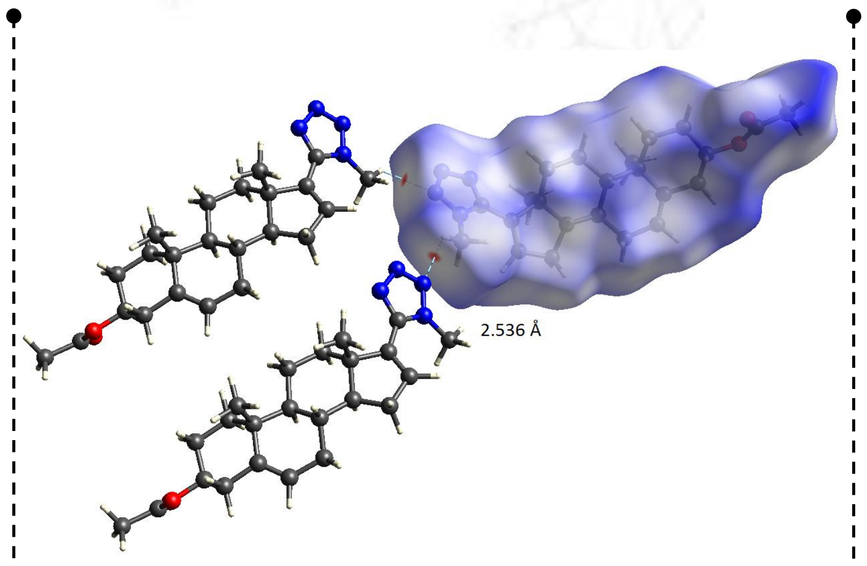


Fig. 1. Hirshfeld surface for conformer **A** of tetrazole **1** mapped with d_{norm} over the range of -0.0673 to 1.8356 and neighboring molecules associated with close contacts.

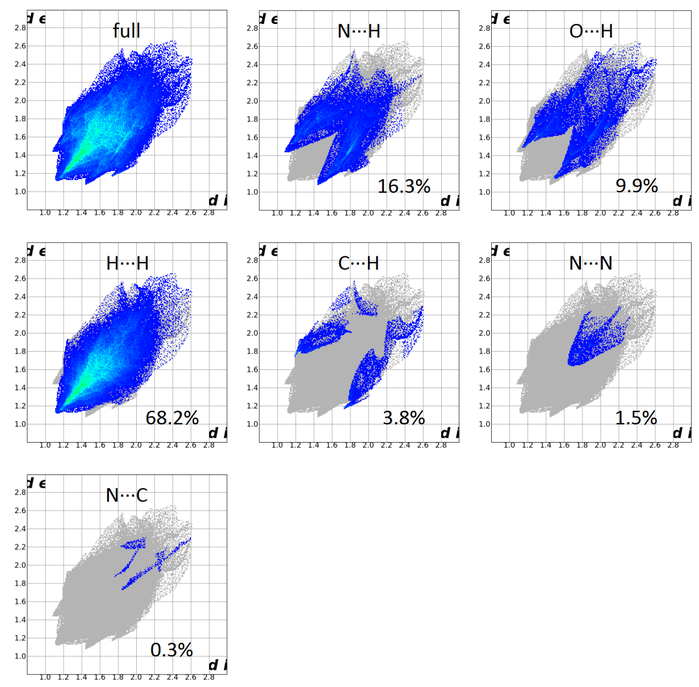


Fig 2. Two-dimensional fingerprint plots of conformer **1A**

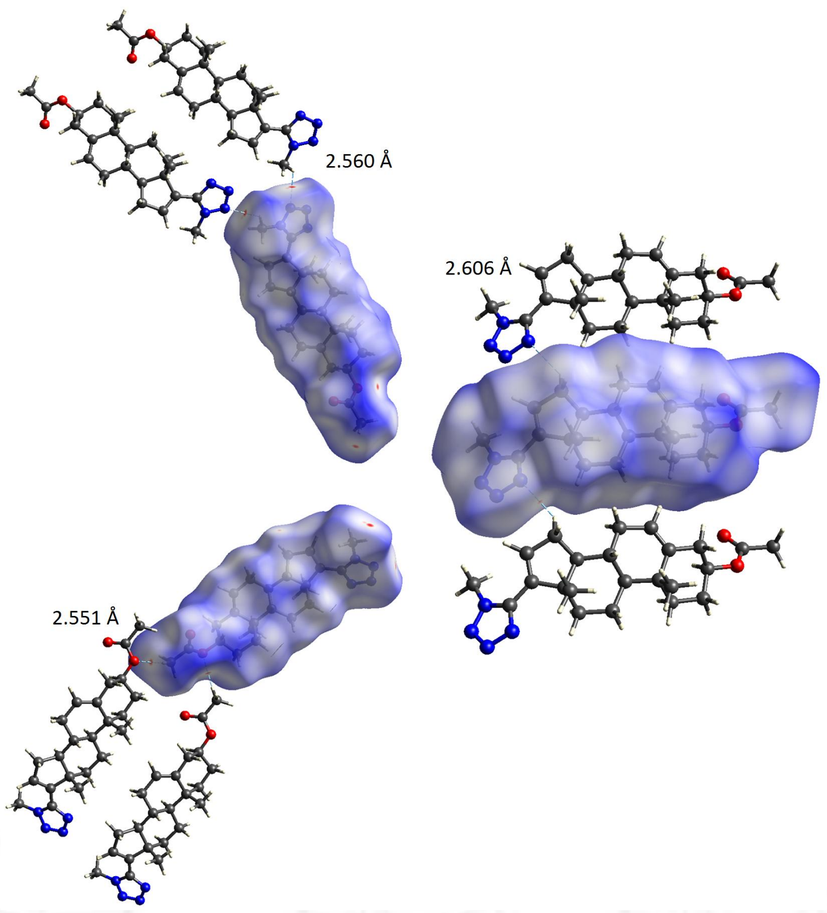


Fig. 3. Hirshfeld surface for conformer **B** of tetrazole **1** mapped with d_{norm} over the range of -0.0462 to 1.7465 and neighboring molecules associated with close contacts.

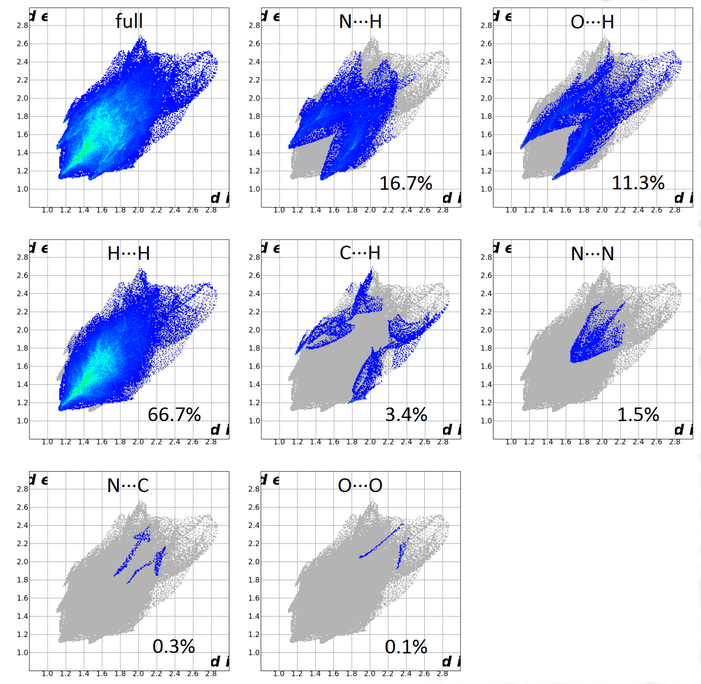


Fig 4. Two-dimensional fingerprint plots of conformer **1B**

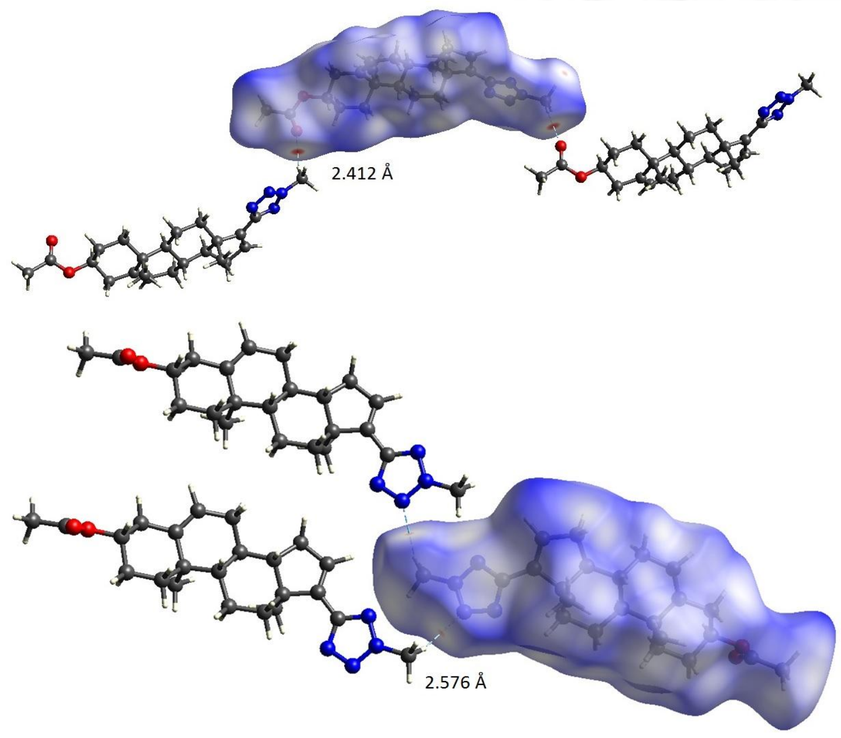


Fig. 5. Hirshfeld surface for tetrazole **2** mapped with d_{norm} over the range of -0.1413 to 1.7329 and neighboring molecules associated with close contacts.

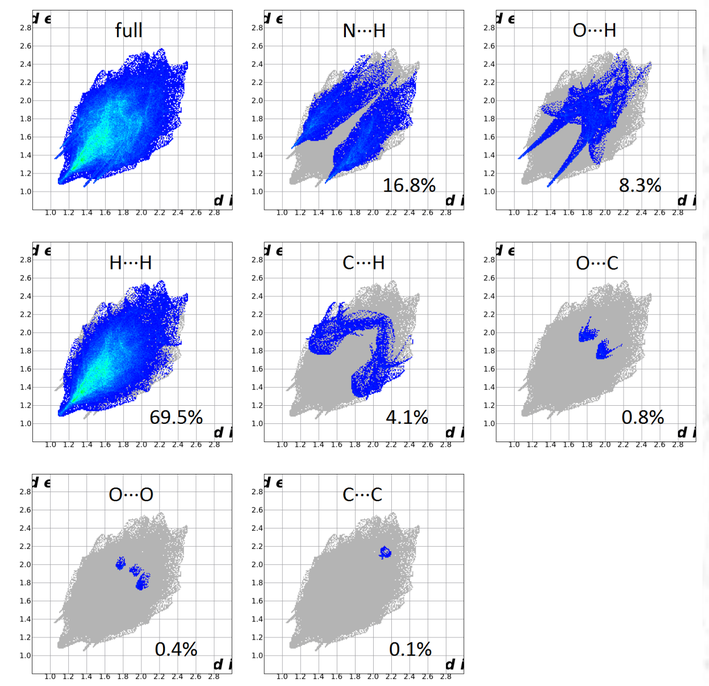


Fig 6. Two-dimensional fingerprint plots of conformer **1A**

Acknowledgement

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