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Synthesis, structural investigations, and anti-cancer activity of new methyl indole-3-carboxylate derivatives

Maria Niemyjska [a], Dorota Maciejewska [a,*], Irena Wolska [b], Paweł Truszkowski [a]

[a] Department of Organic Chemistry, Faculty of Pharmacy, Medical University of Warsaw, 1 Banacha Str., 02 097 Warsaw, Poland
[b] Department of Crystallography, Faculty of Chemistry, Adam Mickiewicz University, 6 Grunwaldzka Str., 60 780 Poznań, Poland

HIGHLIGHTS

- Syntheses of two new bis-indoles were reported.
- Solid-state structures were solved by X-ray or ^{13}C CP/MAS NMR.
- DFT computations were used to propose stable conformation in solid-state.
- Both compounds inhibited human cancer cell lines.

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ABSTRACT

Two new methyl indole-3-carboxylate derivatives: methyl 1-(3′-indolylmethane)-indole-3-carboxylate (**1**), and methyl 1-(1′-benzenosulfonyl-3′-indolylmethane)-indole-3-carboxylate (**2**) were synthesized. They are interesting as the analogs of 3,3′-diindolylmethane, which is intensively tested as a potent anti-tumor agent. Their solid-state structure was characterized using ^{13}C CP/MAS NMR or X-ray diffraction measurements. Molecular modeling was used as a help in the structure elucidation. The solid-state NMR spectroscopy showed only one stable conformer of **1**, but the X-ray diffraction results indicate that compound **2** crystallizes in the triclinic space group $P\bar{1}$ with two molecules, **A** and **B**, in the asymmetric unit. Both compounds inhibited the growth of melanoma, renal and breast cancers cell lines.

1. Introduction

The dietary plants such as cabbage, broccoli, cauliflower and Brussels sprouts produce compounds that can be used as protective agents against tumorigenesis in a variety of human cancers, including breast cancer and prostate cancer [1–3]. Indole-3-carbinol and its acid condensation product 3,3′-diindolylmethane (DIM) are among such compounds. Both compounds are intensively tested in many bioassays [4–9] to elucidate their impact on signal transduction, which leads to cell cycle arrest, apoptosis, down regulation of cancer cell migration, modulation of expression of the CDK inhibitor, $p21^{Cip1/Waf1}$, and stimulation of mitochondrial reactive oxygen species production. Although their activity is well documented, several factors may limit their as chemotherapeutics (due to low bioavailability and promoting of some cancer cells proliferation) [10]. The substitution of indole nitrogen by alkoxy, benzyl and toluenesulfonyl groups enhanced the potency of anti-proliferative properties of indole-3-carbinol [11–13]. These observations suggest that synthetic analogs of DIM substituted at indole nitrogen could also be more potent than DIM which is a very promising compound and can be used as the lead compound to develop new chemotherapeutics with anti-cancer properties. All the facts considered, two new bis-indoles were synthesized which have a methylene linker between the N1 and C3′ atoms (Scheme 1). In this paper, as a part of systematic studies on the structural characterization of bis-indoles [14–16], the synthesis and the spectroscopic characterization of methyl 1-(3′-indolylmethane)-indole-3-carboxylate (**1**) and methyl 1-(1′-benzenosulfonyl-3′-indolylmethane)-indole-3-carboxylate (**2**) were reported. The crystal structure of **2**, and the solid-state analysis of a powder sample of **1** using the ^{13}C CP/MAS NMR method and molecular modeling was also shown.

2. Experimental

2.1. Chemistry

The transformation of methyl 3-indolecarboxylate to bis-indoles **1** and **2** was made using 3-hydroxymethylindole (for **1**) or

* Corresponding author. Tel./fax: +48 22 5720643.
E-mail address: dorota.maciejewska@wum.edu.pl (D. Maciejewska).

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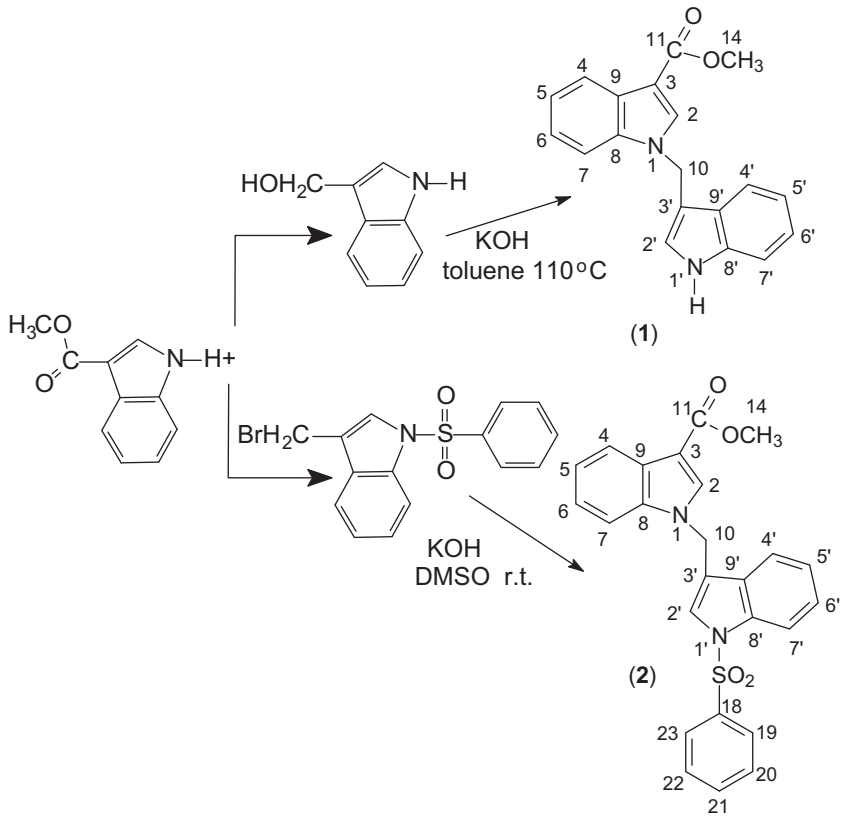

Scheme 1. Syntheses of methyl indole-3-carboxylate derivatives **1** and **2**, their chemical formulas together with the atoms numbering.

1-benzenesulfonyl-3-bromomethylindole (for **2**) as the substrates. 1-Benzenesulfonyl-3-bromomethylindole was synthesized from 1-benzenesulfonyl-3-methylindole as was described earlier [17]. Alkylation of the indole ring at the N atom was reported with reagents such as alcohols in the Pd/Fe_2O_3 catalyzed system [18] or with aldehydes in a benzoic acid catalyzed redox isomerization [19]. The formation of *N*-alkylindoles was also shown for simple alkylhalides [20]. *N*-alkylation of the indole moiety in our experiments was performed by treating equimolar amounts of reagents with sodium in toluene (for **1**) or with potassium hydroxide in dimethylsulfoxide (for **2**) leading to the desired products with good yields. To the best of our knowledge, this is the first report on the 1-(3′-indolylmethane)-indoles. Indispensable chemicals were obtained from major chemicals suppliers as high or highest purity and were used without further purification. Melting points were determined with an Electrothermal 9001 Digital Melting Point Apparatus and are uncorrected. Elemental analyses were performed on a C, H, N, S Elementar GmbH Vario EL III analyzer. IR spectra were recorded on Schimazu FTIR-8300 in KBr tablets.

*2.1.1. Methyl 1-(3′-indolylmethane)-indole-3-carboxylate (**1**)*

Methyl indole-3-carboxylate (1.75 g, 0.01 mol) was dissolved in anhydrous toluene (80 ml) and 3-indolylmethanol (1.77 g, 0.12 mol) in 30 ml anhydrous toluene was added. Before addition of (0.1 g, 0.0043 mol) sodium, 10 ml of the toluene/water azeotrope was removed by distillation. The mixture was stirred at the solvent's boiling temperature for 10 h. To the obtained mixture 3 ml of methanol was added, then 50 ml of water. The organic phase was separated, washed in water and dried with $MgSO_4$. The toluene was evaporated in vacuo. The oily residue of the crude product crystallized after several hours at 4 °C (1.58 g, 51.97%). The crude precipitate was crystallized from ethanol/water (2:1); 0.52 g (17.10%) yield. M.p. 154.5–154.8 °C.-IR (KBr): ν = 3450–3150 (N—H associated), 3120–3000, 3000–2800, 1662 (C=O), 1610, 1535, 1482, 1461, 1446, 1338 (C—N) cm^{-1}. ^1H NMR (299.87 MHz, DMSO) δ = 3.772 (s, 3H, 10-H), 5.607 (s, 2H, 8-H), 6.988 (td, J = 0.9 Hz, 1H, 5′-H), 7.073 (td, J = 1.2 Hz, 1H, 6′-H), 7.190 (td, J_1 = 7.0 Hz, J_2 = 1.0 Hz, 1H, 5-H), 7.241 (td, J_1 = 7.0 Hz, J_2 = 2.0 Hz, 1H, 6-H), 7.367 (bd, J = 8.0 Hz, 1H, 7′-H), 7.516 (bd, J = 8.0 Hz, 1H, 4′-H), 7.594 (d, J = 2.4 Hz, 1H, 2′-H), 7.796 (d, J = 2.0 Hz, 1H, 7-H), 7.969 (d, J = 8.1 Hz, 1H, 4-H), 8.203 (s, 1H, 2-H). ^{13}C NMR (75.41 MHz, DMSO): δ = 41.78 (10-C), 50.62 (14-C), 105.17 (3-C), 109.51 (3′-C), 111.39 (7-C), 111.71 (7′-C), 118.27 (4′-C), 119.03 (5′-C), 120.63 (4-C), 121.42 (6′-C), 121.51 (5-C), 122.30 (6-C), 125.56 (2′-C), 126.19 (9′-C), 126.30 (9-C), 134.93 (2-C), 136.25 (8-C), 136.31 (8′-C), 164.43 (11-C) ppm. $C_{19}H_{16}N_2O_2$ (304.33): calcd. C 74.98, H 5.30, N 9.21; found C 74.98, H 5.38, N 9.21.

*2.1.2. Methyl 1-(1′-benzenosulfonyl-3′-indolylmethane)-indole-3-carboxylate (**2**)*

Methyl indole-3-carboxylate (0.50 g, 0.009 mol) was added to a suspension of potassium hydroxide (0.50 g, 0.009 mol) in 15 ml DMSO. The mixture was stirred at room temperature for 45 min and then 1-benzenesulfonyl-3-bromomethylindole (1.05 g, 0.009 mol) dissolved in 8 ml DMSO was added dropwise. The stirring was continued for 2 h. Then 100 ml of water was added. The obtained beige solid was separated (0.96 g, 72.18%) and crystallized from methanol; 0.58 g bright yellow product (43.61%) yield. M.p. 175.7–176.2 °C. IR (KBr): ν = 3150–3000, 3000–2800, 1692 (C=O), 1533, 1490, 1465, 1446, 1362 (C—N) 1245, 1117 C—O—C, 1218, 1184 (SO_2) cm^{-1}. ^1H NMR (299.87 MHz, DMSO) δ = 3.791 (s, 3H,14-H), 5.638 (s, 2H, 10-H), 7.163–7.259 (m, 3H, 5-H, 5′-H, 6-H), 7.308 (t, J = 8.0 Hz, 1H,6′-H), 7.471 (d, J = 8.0 Hz, 1H, 4′-H), 7.539 (t, J = 7.6 Hz, 2H, 20-H, 22-H), 7.660 (t, J = 7.6 Hz, 1H, 21-H), 7.715 (d, J = 8.0 Hz, 1H, 7-H), 7.907 (d, J = 8.0 Hz, 1H, 7′-H), 7.933 (d, J = 7.6 Hz, 2H, 19-H, 23-H), 7.967 (d, J = 8.0 Hz, 1H, 4-H), 8.133 (s, 1H, 2-H), 8.356 (s, 1H, 2-H). ^{13}C NMR (75.40 MHz, DMSO): δ = 41.31 (10-C), 50.73 (14-C), 105.76 (3-C), 111.37 (7-C), 113.39

(7′-C), 118.21(3′-C), 119.88 (4′-C), 120.71 (4-C), 121.70 (5-C), 122.55 (6-C), 123.70 (5′-C), 125.22 (6′-C), 126.22 (9-C), 126.48 (2′-C), 126.58 (19-C, 23-C), 129.03 (9′-C), 129.78 (20-C, 22-C), 134.61 (8′-C), 134.67 (21-C), 135.42 (2-C), 136.12 (8-C), 136.65 (18-C), 164.32 (11-C) ppm. $C_{25}H_{20}N_2O_4S$ (444.43): calcd. C 67.56, H 4.54, N 6.30, S 7.20; found C 67.54, H 4.52, N 6.30, S 7.05.

2.2. Crystallography

Crystals of **2** suitable for X-ray analysis were grown by slow evaporation from methanol solution. Diffraction data were collected on a KUMA-KM4CCD diffractometer with graphite-monochromated Mo Kα radiation at room temperature. The data were corrected for Lorentz-polarization effects as well as for absorption [21]. The unit cell parameters were determined by least-squares treatment of setting angles of highest-intensity reflections selected from the whole experiment. The structure was solved by direct methods using SHELXS-97 program and refined on F^2 by the full-matrix least-squares method with the SHELXL97 program [22]. The function $\sum w(|F_o|^2 - |F_c|^2)^2$ was minimized with $w^{-1} = [\sigma^2(F_o)^2 + (0.0475P)^2 + 0.4876P]$, where $P = (F_o^2 + 2F_c^2)/3$. All non-hydrogen atoms were refined anisotropically. The coordinates of the hydrogen atoms were generated geometrically and refined 'riding' on their parent atoms with U_{iso} set at 1.2 (1.5 for methyl group) times U_{eq} of the appropriate carrier atom. All details concerning data collection, crystal data and structure refinement are given in Table 1. The Supplementary information in the CIF form is available from Cambridge Crystallographic Database Centre, No. CCDC 878579.

2.3. ^{13}C CP/MAS NMR and molecular modeling

The ^{13}C CP/MAS NMR spectrum of **1** in solid state was recorded with a Bruker Avance DMX 400. The acquisition conditions for ^{13}C CP/MAS NMR at 100.62 MHz were: pulse duration, 4.5 μs; contact time, 4 ms; repetition time, 20 s; spectral width, 24 kHz; number of transients, 1000; spinning speed, 8 kHz. ^1H and ^{13}C NMR, HSQC and HMBC spectra in solution were recorded at 25 °C with a Varian NMRS-300 spectrometer and standard Varian software was employed. Chemical shifts δ (ppm) for ^{13}C were referenced to TMS.

The optimized atom coordinates of **1** were used for computation of shielding constants σ (ppm) of ^{13}C atoms as a help in assignment of resonances in the solid-state NMR spectrum, and structural analysis. We employed the DFT method with B3LYP/6-311(d,p) hybrid functional for structure optimization, and the CPHF–GIAO approach for the NMR shielding constants computations using Gaussian 09 program [23].

Table 1
Crystal data, data collection and structure refinement for compound **2**.

Compound	**2**
Empirical formula	$C_{25}H_{20}N_2O_4S$
Formula weight	444.49
T (K)	293(2)
Wavelength (Å)	0.71073
Crystal system, space group	Triclinic, $P-1$
Unit cell dimensions	
a (Å)	11.2632(7)
b (Å)	12.4373(8)
c (Å)	16.5899(7)
α (°)	80.409(4)
β (°)	83.086(4)
γ (°)	69.724(6)
Volume (Å^3)	2144.6(2)
Z, D_x (Mg/m^3)	4, 1.377
μ (mm^{-1})	0.187
$F(000)$	928
θ range for data collection (°)	4.10–25.35
hkl range	$-13 \le h \le 9$ $-14 \le k \le 14$ $-19 \le l \le 19$
Reflections	
Collected	13,645
Unique (R_{int})	7806 (0.020)
Observed ($I > 2\sigma(I)$)	5600
Data/restraints/parameters	7806/0/577
Goodness-of-fit on F^2	1.032
$R(F)$ ($I > 2(I)$)	0.0462
$wR(F^2)$ (all data)	0.1176
Max./min. $\Delta\rho$ (e/Å^3)	0.238/−0.351

2.4. Cytotoxicity against human cancer cell lines

The in vitro cell line screening was carried out in The National Institute of Health, Bethesda, USA as a part of the DTP anti-cancer drug discovery program. The evaluation against 60 human tumor cell lines including leukemia, melanoma, and non-small cell lung, colon, CNC, ovarian, renal, prostate, breast cancers was made at a single dose of 10 μM. Detailed information about the methodology and the screening services are available at the website http://dtp.nci.nih.gov.

3. Results and discussion

3.1. X-ray analysis of **2**

The molecular and crystal structure of methyl 1-(1′-benzenosulfonyl-3′-indolylmethane)-indole-3-carboxylate (**2**) in solid

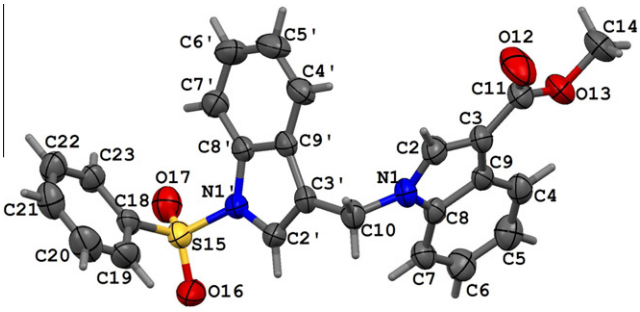

Fig. 1. The displacement ellipsoid representation of the molecule **B** of **2**.

Table 2
Selected bond lengths (Å) and angles (°) and torsional angles (°) for **2.**

	2A	**2B**
N1—C2	1.349(3)	1.358(3)
N1′—C2′	1.398(3)	1.400(3)
N1—C8	1.387(2)	1.387(3)
N1′—C8′	1.417(3)	1.405(3)
C2—C3	1.365(3)	1.368(3)
C2′—C3′	1.338(3)	1.339(3)
C8—C9	1.405(3)	1.411(3)
C8′—C9′	1.394(3)	1.403(3)
N1—C10	1.461(3)	1.462(3)
N1′—S15	1.655(2)	1.672(2)
C8—N1—C10	126.0(2)	126.0(2)
N1—C8—C7	130.0(2)	130.0(2)
N1—C10—C3′	113.1(2)	113.0(2)
C8′—N1′—S15	126.2(2)	124.7(1)
N1′—S15—C18	106.7(1)	103.9(1)
C2′—N1′—S15—C18	−75.7(2)	92.2(2)
N1′—S15—C18—C19	−97.5(2)	99.2(2)
N1—C10—C3′—C2′	111.3(2)	115.5(2)
C7′—C8′—N1′—S15	26.3(3)	−21.8(3)
C2—C3—C11—O12	−3.3(4)	3.7(4)
C2—C3—C11—O13	177.1(2)	−174.8(2)
C3—C11—O13—C14	−179.5(2)	178.8(2)

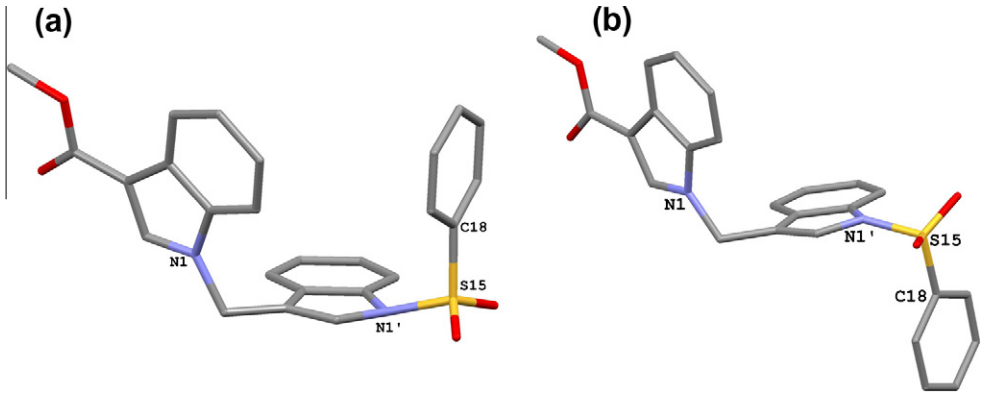

Fig. 2. The arrangement of particular fragments of the molecules **A** and **B** together with the numbering of atoms essential for the conformational analysis.

state were analyzed by single crystal X-ray diffraction. The results indicate that the compound crystallizes in the triclinic $P\bar{1}$ space group with two independent molecules, **A** and **B,** in the asymmetric unit cell. The atomic numbering scheme, for a clarity only for molecule **B**, is shown in Fig. 1 (the drawings were performed with The Mercury Program [24]). The structure consists of two indole rings with the methylene group joined N1 and C3′ atoms, benzenosulfonyl substituent at N1′ and methylester at C3. Selected bond lengths, bond angles and torsion angles are listed in Table 2. As we can see only the N1′A—S15A and N1′B—S15B bond lengths vary slightly, but some torsion angles reveal important dissimilarities. In consequence, in the molecules **A** and **B** the principal differences lie in the steric arrangement of the indole system N1′···C9′ and the benzenosulfonyl group (Fig. 2). Different arrangement of these fragments can be described by the torsion angle C2′—N1′—S15—C18 as well as N1′—S15—C18—C19 (Table 2). The indole frameworks are essentially planar and their planes make an angle of 74.78(4)° and 82.17(4)° in **A** and **B**, respectively. The orientation the ester group at C3 with respect to the indole (N1···C9) part can be described by the appropriate torsion angles (Table 2). In the crystals, intermolecular C—H···O and C—H···N hydrogen bonds were found to be the main force which determines the crystal packing of molecules. The geometric parameters of all these bonds are given in Table 3. The packing arrangement is shown in Fig. 3. The crystal structure is built of layers of the molecules **A** (red) and **B** (blue[1]), alternately, parallel to (0 1 0) plane. Within the layers the molecules are connected via C5···O17, C6···O16, C6′···O12 hydrogen bonds for **A** and C2··· O12, C20···N1 for **B**. Then the layers interact via C2′ B···O12A, C2A···O16B contacts and van der Waals interactions.

3.2. Solid-state structure of **1** based on ^{13}C CP/MAS NMR spectrum

So far, it was impossible to obtain single crystals of **1** suitable for X-ray diffraction measurements. To analyze the solid-state structure of **1**, we decided to employ the ^{13}C CP/MAS NMR method for the analysis of a powder sample of **1**. Solid-state NMR spectroscopy is broadly used to characterize pharmaceutical solids and compounds which are produced during the drug development process. The correlation between the ^{13}C experimental resonances and the theoretical values of the shielding constants obtained for the corresponding structure pointed to the stable conformation present in the solid state [25]. The solid-state ^{13}C CP/MAS NMR spectrum of **1** is shown in Fig. 4. We have observed the peak multiplicity of resonances in agreement with the solution-like spectrum which means that only a single structure is detected in the solid state. It is unlike the single-crystal structure of **2** in which two independent molecules, **A** and **B,** in the asymmetric unit cell are detected (see Section 3.1). To propose the solid-state structure of **1** we have analyzed two conformations of **1**, named **1a** and **1b** (see Fig. 5,Tables 1S and 2S in Supplement) which differ in the torsion angle C1—C10—C3′—C9′ (−61.4° and 71.5° for **1a** and **1b**, respectively). Geometry calculations were made at DFT level with B3LYP/6-311(d,p) hybrid functional. We found that both conformations are almost energetically equivalent. The conformation **1b** is slightly preferred by 2.5 kJ/mol but this difference could not be decisive for the preferred structure in the solid state where the intermolecular interactions play a significant role. Thus, we computed the NMR shielding constants σ (ppm) for the atomic coordinates corresponding to both conformations, and compared them with the experimental chemical shifts δ (ppm). Next, we analyzed the correlation coefficients R^2 for the linear function $\delta = f(\sigma)$ calculated for both conformers (see Fig. 1S in Supplement). On the basis of the higher correlation coefficient ($R^2 = 0.996$) and by comparison with the conformation of **2**, we propose the structure of **1a** as favored in the solid state. The packing mode of molecules is stabilized by quite strong intermolecular hydrogen bonds, which shifts (as compared with the solution spectrum) downfield the resonance of C11 carbonyl atom ($\Delta\delta = 2.6$ ppm), and simultaneously shifts upfield the resonances of C5 and C2 atoms ($\Delta\delta = -0.6$ and −0.9 ppm, respectively). The ester group is located in the plane of the indole ring which is characterized by the C9—C3—C11—O12 torsion angle equal to 179.9°, and downfield shift of C4 atom resonance by 2.2 ppm.

Table 3
Hydrogen bonding geometry (Å and °) for **2**.

D—H···A	*d*(D—H)	*d*(H···A)	*d*(D···A)	<(DHA)
C5A—H5A···O17A^{i}	0.93	2.72	3.634(3)	169
C6A—H6A···O16A^{ii}	0.93	2.61	3.426(3)	146
C6′A—H6′A···O12A^{iii}	0.93	2.48	3.398(3)	170
C20B—H20B···N1B^{i}	0.93	2.69	3.576(3)	160
C2B—H2B···O12B^{iv}	0.93	2.53	3.310(3)	142
C2A—H2A···16B^{iii}	0.93	2.37	3.269(3)	163
C2′B—H2′B···O12A^{iii}	0.93	2.30	3.170(3)	155

Symmetry codes: (i) $-x+2, -y, -z$; (ii) $x, y+1, z$; (iii) $x, y-1, z$; (iv) $x-1, y, z$; (v) $-x, -y, -z+1$.

3.3. Cytotoxicity in preliminary anti-cancer assay

Compounds **1** and **2** were accepted for evaluation in the DTP anti-cancer drug discovery program (National Cancer Institute, Bethesda, USA) in a 60-cell line panel. Detailed information is

[1] For interpretation of color in Figs. 1–3 and 5, the reader is referred to the web version of this article.

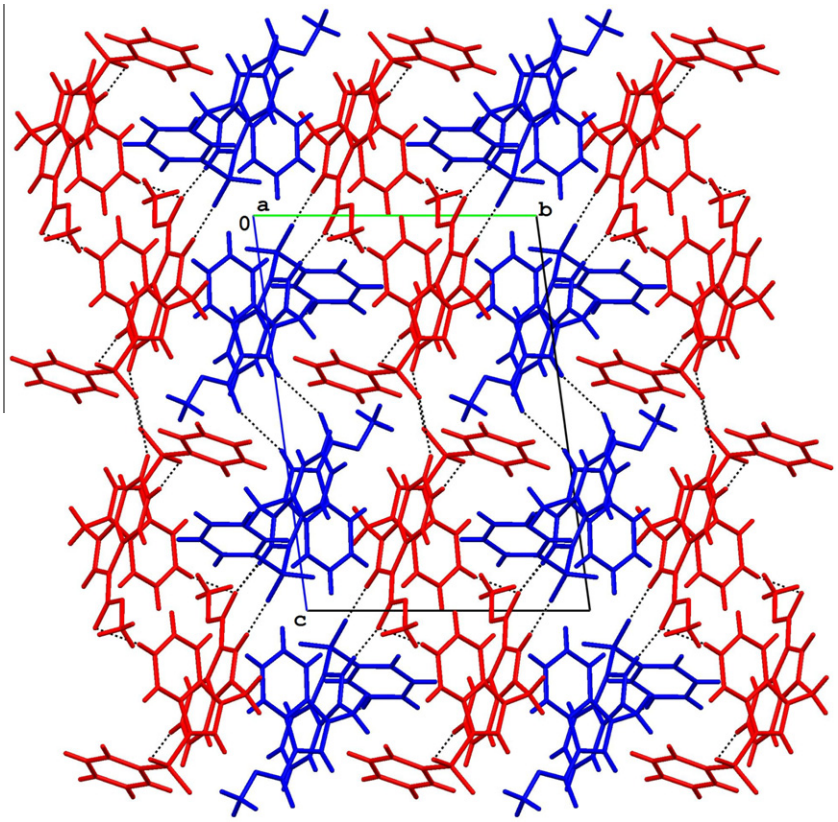

Fig. 3. Projection of the crystal structure of **2** along the *a* axis with layers of the molecules **A** (red) and **B** (blue), alternately.

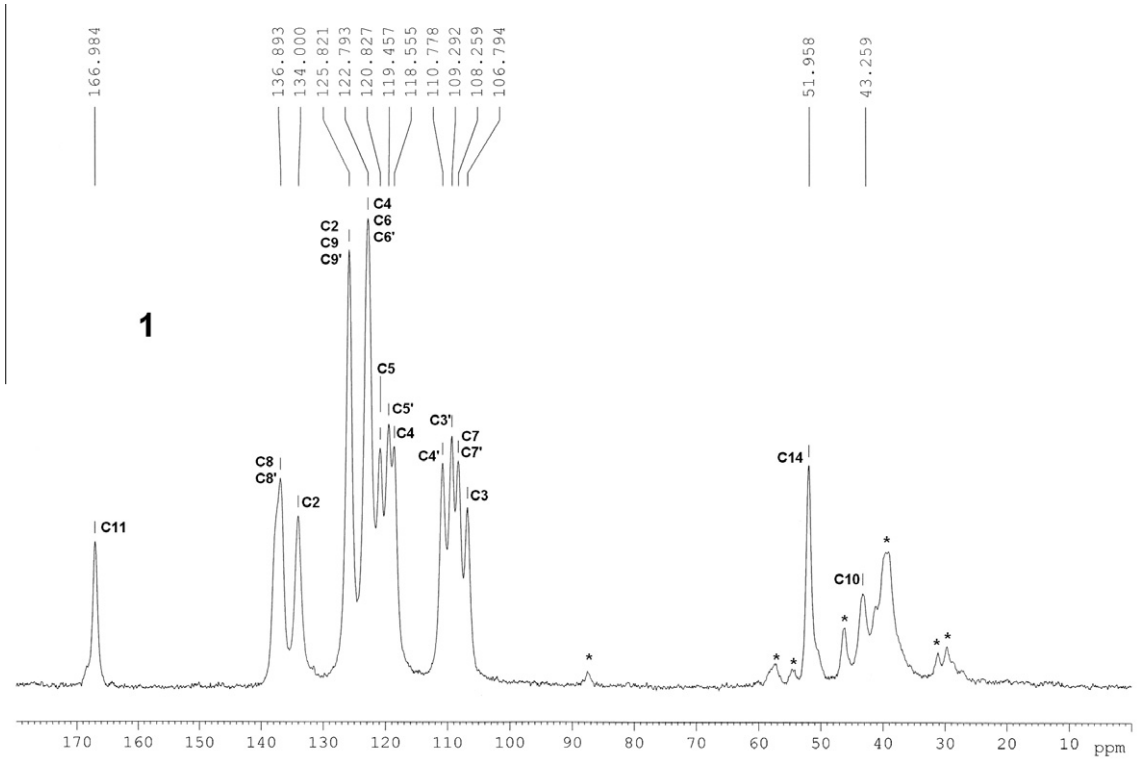

Fig. 4. ^{13}C CP/MAS NMR spectrum of **1** in solid state. Sidebands are marked with an asterisk.

available at the website http://dtp.nci.nih.gov. Full results are shown in Figs. 2S and 3S in the Supplement.

Both compounds inhibited the growth of the treated cells as compared with the untreated control cells of melanoma, renal

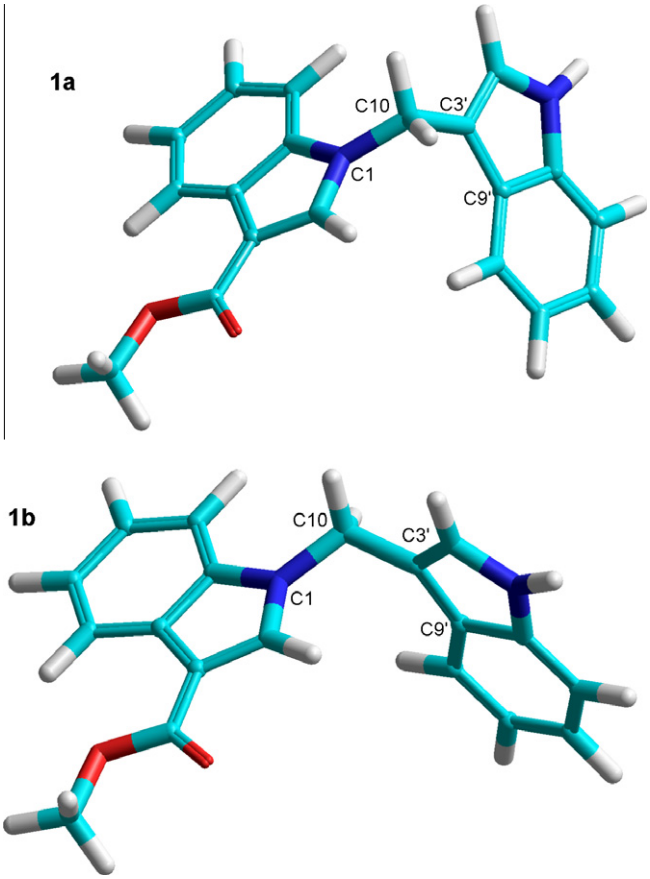

Fig. 5. The analyzed conformations of **1**.

and breast cancers. Substitution of the N atom by the benzenesulfonyl group gave a compound that was more potent relative to the unsubstituted analog. The percentage of inhibition was highest for the T-47D breast cancer line, and was equal to 50.9% for **2**. Compound **2** inhibits the growth of CAKI-1 renal cancer line and UACC-62 melanoma in 50.4% and 47.1%, respectively. Compound **1** is less active and inhibits the growth of MDA-MB-435 melanoma, CAKI-1 and T-47D in 48.4%, 41.5% and 31.5%, respectively.

4. Conclusions

Two new bis-indoles methyl 1-(3′-indolylmethane)-indole-3-carboxylate (**1**) and methyl 1-(1′-benzenosulfonyl-3′-indolylmethane)-indole-3-carboxylate (**2**) were synthesized in sufficient yields. Both compounds inhibited the growth of melanoma, renal and breast cancers cell lines at a dose of 10 μM. Substitution of N atom by the benzenesulfonyl group gave a compound that was more potent relative to the unsubstituted analog.

Their solid-state conformations were successfully established. The X-ray diffraction studies showed that compound **2** crystallizes in the triclinic space group $P\bar{1}$ with two independent molecules, **A** and **B,** in the asymmetric unit cell. The principal differences in the molecules **A** and **B** were found to lie in the steric arrangement of the indole system N1′···C9′ and the benzenosulfonyl group. The three-dimensional supramolecular structure results from the combination of the C—H···O and C—H···N interactions.

Single resonances in the ^{13}C CP/MAS NMR spectrum of **1** indicate that only one conformer is present in the solid state. The packing mode of molecules is stabilized by quite strong intermolecular hydrogen bonds between C11—O and C5—H or C2—H groups.

Acknowledgments

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molstruc.2012.05.031.

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