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Ab initio and Semiempirical Conformational and Configurational Analysis of *N*-2-(1,4-Dioxane)-*N′*-(4-methylbenzenesulfonyl)-*O*-(4-methylphenoxy)isourea

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The conformational, configurational behavior and the structure of *N*-2-(1,4-dioxane)-*N′*-(4-methylbenzenesulfonyl)-*O*-(4-methylphenoxy) isourea **1** has been studied using *ab initio* and semiempirical calculations (AM1 and PM3). The *endo*-anomeric effect and hydrogen bonds control the population of dioxane ring conformers or anomers but not the configuration interconversion of the imine of imidoyl moiety. *Ab initio* and AM1 and PM3 calculations demonstrate that the dioxane ring adopts a chair conformation, that the imidoyl amino group prefers an axial conformation and that the tosyl and tolyl groups about the C=N bond retain an *E* configuration. The computational analysis of **1** complements the X-ray findings.

Keywords: Anomeric effect, Conformational analysis, Configurational analysis, *ab initio*, AM1, PM3

INTRODUCTION

The important role of pyranose in biology has led to theoretical and experimental investigation to understand the factors that affect its conformation, relative rotamer stabilities, and anomeric abundance (the most significant energetic stabilizing factor in sugar chemistry) [1]. In recent years, the anomeric effect and the conformational analysis of 1,4-dioxane and its substituted analogs has attracted much attention [2-42]. The major area of interest has been the pharmaceutical activities of 1,4-dioxane [27-31]. The anomeric effect is well recognized as an important factor in defining the predominant conformational state of many cyclic heteroatom-containing compounds. The conformational geometry of the transition and/or intermediate states of these compounds has been documented to establish the selectivity of the chemical reactions and/or the stereochemistry of their adducts [43-45]. Since it is entirely conceivable that the pharmaceutical activity of dioxane is related to the physicochemical properties, thorough investigation of 1,4-dioxane derivatives was initiated many years ago [2-42].

Chapman and Hester analyzed the inversion of 1,4-dioxane by *ab initio* molecular orbital theory at the HF/6-31G* and BLYP/6-31G* levels. They concluded that the chair conformation is the lowest energy state, followed by the two twist-boats [21].

Recently, Dabbagh and coworkers studied the structure, conformation of 1,4-dioxane, configuration of the imine group of the imidoyl moiety and the anomeric effect of *N*-2-(1,4-dioxane)-*N′*-(4-methylbenzenesulfonyl)-*O*-(4-methylphenoxy) isourea (**1**) using X-ray crystallographic analysis, Scheme 1 [2].

The purpose of the present paper is to provide evidence that *ab initio* and semiempirical calculations (AM1 and PM3) are reliable methods to investigate the anomeric effect and structure of **1**. The second aim of this report is to establish that

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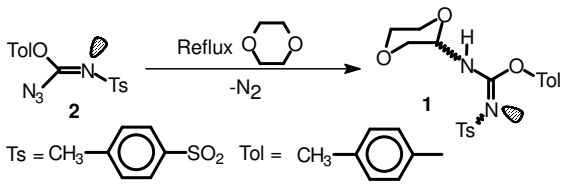


Scheme 1. Synthesis of compound **1** from the thermal decomposition of **2** (imidoyl azide).

the large imidoylamino group [(4-CH_3-C_6H_4-O-C=N-SO_2-C_6H_4-CH_3-4)-NH-] adopts an axial position (the anomeric effect). Additional aims of this report are to investigate the factors (hydrogen bonds and steric hindrance to the anomeric effect) that contribute to this axial preference and to study the conformations and configurations of **1**, Scheme 1.

We believe this system, allows us to shed more light on the phenomenon known as the anomeric effect.

EXPERIMENTAL

Synthesis

Compound **1** was synthesized by the thermal decomposition of *N'*-(4-methylbenzenesulfonyl)-*O*-(4-methylphenoxy)imidoyl azide (**2**) in refluxing dioxane, diagrammed in Scheme 1, as previously published [46].

Computational Analysis

Ab initio, AM1 and PM3 calculations of **1** were performed by HyperChem-V.7.0, using the Polak-Ribiere algorithm.

The energy of all conformers was calculated by rotation about the O4-C31-N2-C3 angle. The conformations and configurations of $\mathbf{1}\boldsymbol{E}_{\mathrm{axA1}}$ were calculated by X-ray and computational analysis.

RESULTS AND DISCUSSION

Computational Analysis

On the basis of the X-ray data (Fig. 1, Scheme 2, and Tables 3 and 4) the assumption was made that the imino amino group (-NHG) has the most stable axial conformer (due to anomeric effect and intramolecular hydrogen bonds).

The calculation of the energy of all conformers by rotation about O4-C31-N2-C3 angle was an assumption that turned out to be correct. The conformations and configurations of $\mathbf{1}\boldsymbol{E}_{\mathrm{axA1}}$, the most stable conformer as calculated by X-ray and computational analysis, are shown in Fig. 1, Table 1, and Schemes 2 and 3. The axial conformer $\mathbf{1}\boldsymbol{E}_{\mathrm{ax}}$ calculated by

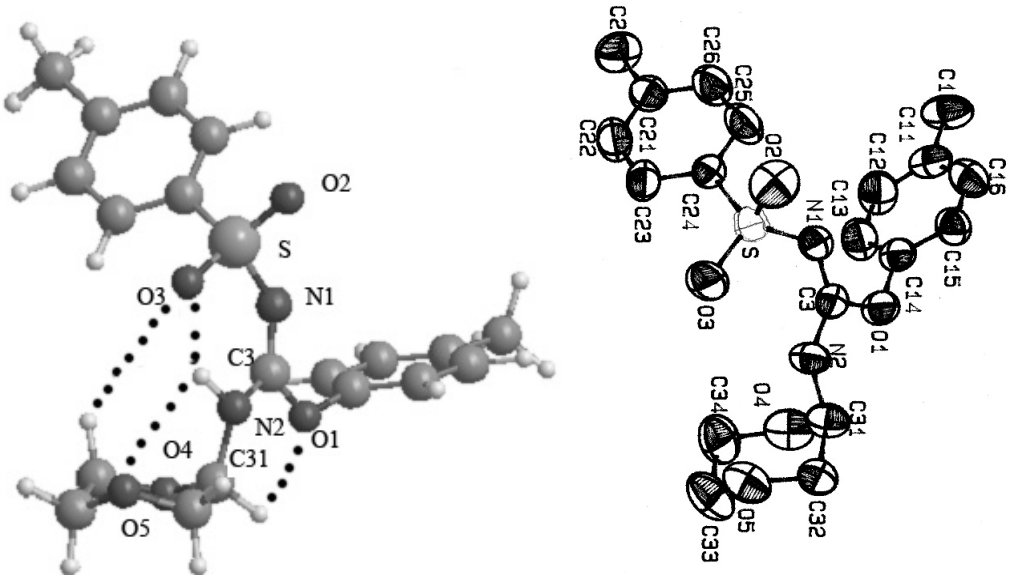


Fig. 1. Molecular structure and hydrogen bonding (**…..**) for **1** optimized by *ab initio* (left). Molecular structure of **1** from X-ray crystallographic analysis (right).

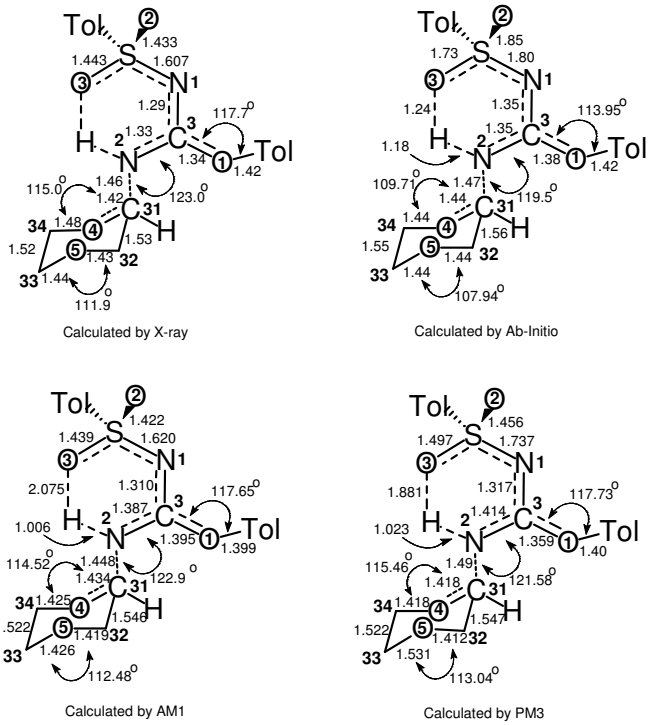
Table 1. Relative Energy, Heat of Formation, Dipole Moment and Dihedral Angle for **1**[a]

Entry	Conformer	Angle of O4-C31-N2-C3	ΔH_f (kcal mol^{-1})	μ (D)	$E_{rel.}$ (kcal mol^{-1})[b]
1	**1*E***$_{ax}$A$_1$[b]	-82.35	-111.85	6.190	0
2	**1*E***$_{ax}$A$_2$	-90.0	-111.83	6.148	0.02
3	**1*E***$_{ax}$A$_3$	-78.0	-111.82	6.219	0.03
4	**1*E***$_{ax}$A$_4$	-73.53	-111.62	6.257	0.23
5	***1*E***$_{ax}$***B*$_1$	-155.66	-109.97	5.981	1.80
6	**1*E***$_{ax}$B$_2$	179.59	-108.36	5.941	3.49
7	**1*E***$_{ax}$B$_3$	150.26	-104.73	6.045	7.12
8	**1*E***$_{ax}$C$_1$	60.0	-104.44	6.285	7.41
9	**1*E***$_{ax}$C$_2$	86.35	-104.40	6.304	7.45
10	**1*E***$_{eq}$D$_1$	94.59	-106.53	5.979	5.32
11	**1*E***$_{eq}$D$_2$	60.0	-104.71	5.976	7.14
12	**1*E***$_{eq}$D$_3$	30.23	-102.45	6.276	9.4
13	**1*E***$_{eq}$E$_1$	-58.87	-106.55	6.661	5.30
14	**1*E***$_{eq}$E$_2$	-89.83	-105.18	6.697	6.67
15	**1*E***$_{eq}$E$_3$	-30.33	-104.43	6.664	7.42
16	**1*E***$_{eq}$F$_1$	160.0	-106.13	6.155	5.72
17	**1*E***$_{eq}$F$_2$	175.65	-105.90	6.23	5.95
18	**1*E***$_{eq}$F$_3$	-160.21	-104.58	6.384	7.27
19	**1*Z***$_{ax}$A$_1$[c]	-72.34	-94.57	4.944	17.28
20	**1*Z***$_{ax}$A$_2$	-155.53	-93.06	5.453	18.79
21	**1*Z***$_{ax}$A$_3$	83.46	-86.14	5.380	25.71
22	**1*Z***$_{ax}$B$_1$	-70.74	-98.44	8.009	13.41
23	**1*Z***$_{ax}$B$_2$	-82.18	-97.07	7.936	14.78
24[d]	**1**-NMe***E***$_{ax}$	-78.95	-109.21	6.726	2.64
25[d]	**1**-NMe***E***$_{eq}$	-53.11	-104.89	6.784	6.96

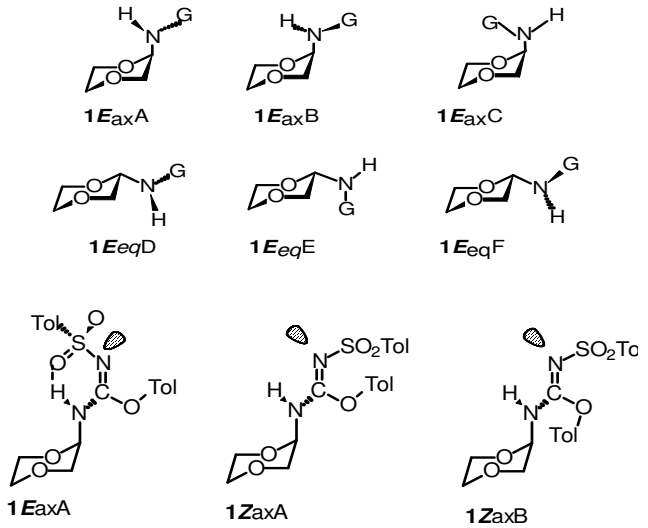
[a]Calculated by AM1, see Scheme 3. [b]Erel. = $\Delta\Delta H_f$. [b]*E* = *E*-isomer. [c]*Z* = *Z*-isomer. [d]Compound 1 (*N*-methylated).

AM1 is 5.3 kcal mol^{-1} more stable than the equatorial **1*E***$_{eq}$ (see Table 1 and Scheme 3). As shown in Table 3, the relative energy of the optimized geometries of **1*E***$_{ax}$ over **1*E***$_{eq}$ calculated by STO-3G (19.50 kcal mol^{-1}) and PM3 (8.20 kcal mol^{-1}) also indicates that **1*E***$_{ax}$ is the more stable conformation. The *E* isomer **1*E***$_{ax}$A1 (of the imidoyl group -C=N-) is 13.0 kcal mol^{-1} more stable than the *Z*-isomers (**1*Z***$_{ax}$B).

Other *Z*-isomers (**1*Z***$_{ax}$A$_1$, **1*Z***$_{ax}$A$_2$, **1*Z***$_{ax}$A$_3$, **1*Z***$_{ax}$B$_2$) are less stable than **1*Z***$_{ax}$B$_1$, as shown in Table 1. The results of this investigation are consistent with MM2 and *ab initio* [25,27, 32,47-56] and several reported semiempirical calculations [31,47-49,51,56] in defining the anomeric effect. The next step was to compare the findings of the *ab initio,* AM1 and PM3 calculations with the experimental-based values obtained by X-ray, Fig. 1, Scheme 2 and Tables 2, 3, and 4. Computational analysis complements the experimental findings. The only discrepancy was the length of the O4-C31 bond (which indicates the extent of the anomeric effect) calculated by AM1 (1.43 Å) versus *ab initio* (1.44 Å) for the dioxane ring of 1. This bond length was longer than the value obtained by X-ray



Scheme 2. Selected bond lengths and bond angles calculated by *ab initio*, AM1, PM3 and X-ray methods. For simplicity selected atoms are numbered.



Scheme 3. Conformation and configurations of compound **1**.

(1.42 Å). In this case, the value (1.418 Å) calculated by PM3 matches the X-ray value. Thus, it appears that *ab initio,* AM1 and PM3 are reliable methods for optimizing the geometry of larger molecules. However, certain calculated bond lengths and bond angles may deviate from X-ray values (depending on the method of calculation).

The second issue raised here was the determination of the major forces influencing the preference for the axial *N*-amination (*via* imidoyl nitrene) of 1,4-dioxane.

Evaluation of Hyperconjugative Effect

Generally the imidoyl azides do not insert into the C-H bonds Jones and Kirby have used, increasing electron demand on oxygen as a probe to test the relationship between bond length and reactivity in tetrahydropyranyl acetates and phosphate monoester dianions, Scheme 4 [57-59]. In the case of **1**, hyperconjugation of the oxygen lone pairs of 1,4-dioxane increases the reactivity of the C-H bond forcing dioxane into a planar conformation. This would allow the nitrene or azide to add to the axial side producing conformer $\mathbf{1}\boldsymbol{E}_{ax}$. The formation of conformer $\mathbf{1}\boldsymbol{E}_{eq}$ is blocked by electronic repulsion, as seen in Scheme 5. The axial conformer $\mathbf{1}\boldsymbol{E}_{ax}$ is favored in solid phase or in non-polar solvents [1,42]. In other words, *ab initio,* AM1 and PM3 confirmed the axial preference. Strong intramolecular hydrogen bonds exist between the N-H with oxygen (O3) of S=O (-N-H·····O=S-) of the tosyl group, the NH and oxygen (O4) lone pairs of dioxane, and the oxygen (O1) of the tolyl group with the anomeric hydrogen (C31-H). The *endo*-anomeric effect of the oxygen of dioxane and these strong intramolecular hydrogen bonds push the G-NH to hold the axial position.

Evaluation of Steric Hindrance Effect

Initially, we expected the bulky imidoyl nitrene to choose the equatorial position of the dioxane ring (Reverse Anomeric Effect, RAE) and steric hindrance to overcome the anomeric effect. However, the relative energy calculated by *ab initio*, AM1 and PM3 indicated that $\mathbf{1}\boldsymbol{E}_{ax}$ is more stable than $\mathbf{1}\boldsymbol{E}_{eq}$, see Tables 2 and 5. The X-ray and ^{1}H NMR of 1 also confirmed that the anomeric effect overcame the large steric hindrance [1,42]. This indicates that there is no RAE due to steric hindrance. In fact, the bulky imidoyl group retains a proper axial position, which minimizes any repulsion by the

Table 2. Comparison of the Total Energy of Optimized Geometry of $\mathbf{1}\boldsymbol{E}_{ax}$ and $\mathbf{1}\boldsymbol{E}_{eq}$

Method of calculation	Total energy (kcal mol^{-1})		ΔE_T[a]
	$\mathbf{1}\boldsymbol{E}_{ax}$	$\mathbf{1}\boldsymbol{E}_{eq}$	
AM1	-114023.5	-114017.0	6.5
PM3	-105344.7	-105336.5	8.2
STO-3G	-999708.5	-999689.0	19.5

[a] $\Delta E_T = E_{\mathbf{1}\boldsymbol{E}eq} - E_{\mathbf{1}\boldsymbol{E}ax}$.

Table 3. Comparison of Selected Experimental and Calculated Bond Lengths of **1**

Bond type	X-Ray	*Ab initio*	AM1	PM3
O4-C31	1.42	1.44	1.43	1.42
N2-C31	1.46	1.47	1.45	1.49
N2-C3	1.33	1.35	1.39	1.41
O1-C3	1.34	1.35	1.40	1.36
N1-C3	1.29	1.35	1.30	1.32
S-N1	1.61	1.80	1.62	1.74
S-O3	1.44	1.73	1.44	1.50

Table 4. Comparison of Selected Experimental and Calculated Bond Angles of **1**

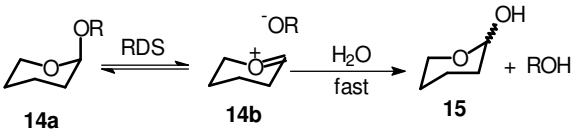
Bond angle type	X-Ray	*Ab initio*	AM1	PM3
C31-O4-C34	115.0	109.7	114.5	115.5
C31-N2-C3	123.0	119.5	122.9	121.6
C32-O5-C33	111.9	107.9	112.5	113.0
C3-O1-C14[a]	117.7	114.7	117.7	117.7

[a] C14 = carbon of phenyl ring of tolyl.

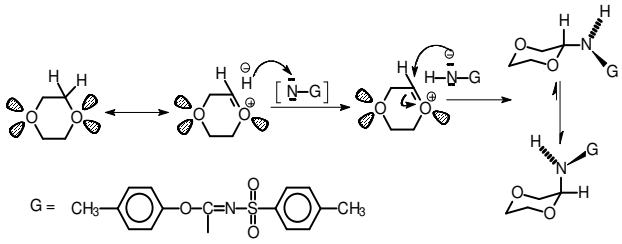
Table 5. Comparison of the Total Energy, Heat of Formation, Dipole Moment and Dihedral Angle for Optimized Geometry of $\mathbf{1}\boldsymbol{E}_{ax}$

Method of calculation	Angle of O4-C31-N2-C3	ΔH_f (kcal mol^{-1})	μ (D)	Total energy (kcal mol^{-1})[a]
AM1	-76.2	-116.9	6.706	-114023.5
PM3	-67.4	-129.8	5.450	-105344.7
STO-3G	-79.9	–	4.710	-999708.5

[a] $\boldsymbol{E}$ = *E*-isomer.



Scheme 4. Hyperconjugative effect of oxygen lone pairs of tetrahydropyranyl.



Scheme 5. Hyperconjugative effect of oxygen lone pairs of 1,4-dioxane.

protons of the dioxane ring, see Figures 1 and 2.

Evaluation of Hydrogen Bonding Effect

The preferred axial conformation of **1** was initially presumed to be due to several intramolecular hydrogen bonds in addition to the anomeric effect (Figs. 1 and 2). AM1 analysis of *N*-methylated **1** shows that **1**-NH***E***$_{ax}$ is 2.64 kcal mol^{-1} more stable than **1**-NMe***E***$_{eq}$. This indicates that the hydrogen bonds add 2.64 kcal mol^{-1} to the stability of the axial conformer, assuming the steric effect of the methyl group is negligible when compared to the very large imidoyl moiety (see Table 1 and Fig. 2).

CONCLUSION

The anomeric effect (hyperconjugative effect) plays a major role in the axial preference of the imidoyl amino group. The electronic effects (resonance, induction, hydrogen bonds) have secondary roles in the axial preference of HNG (G = *4*-Me-C_6H_4-O-C=N-SO_2-C_6H_4-Me-*p*). Hydrogen bonds contribute 2.64 kcal mol^{-1} to the axial preference of HNG. Steric hindrance plays a minor role in the RAE. The *endo*-anomeric effect and hydrogen bonds are responsible for the preferred axial conformation and *E*-configuration of **1** in solid phase. *Ab initio*, AM1 and PM3 calculations of **1** complement the X-ray findings and demonstrate that the dioxane ring adopts a chair conformation, that the imidoyl amino group prefers an axial conformation and that the tosyl and tolyl groups about the C=N bond retain an *E* configuration. We have recently reported a similar phenomenon for compound **2** [60]. The results of this investigation demonstrate that *ab initio*, AM1 and PM3 calculations are consistent with the findings of MM2 and X-ray in defining the anomeric effect, conformation, configuration and structure of *N*-2-(1,4-dioxane)-*N'*-(4-methylbenzenesulfonyl)-*O*-(4-methylphenoxy) isourea.

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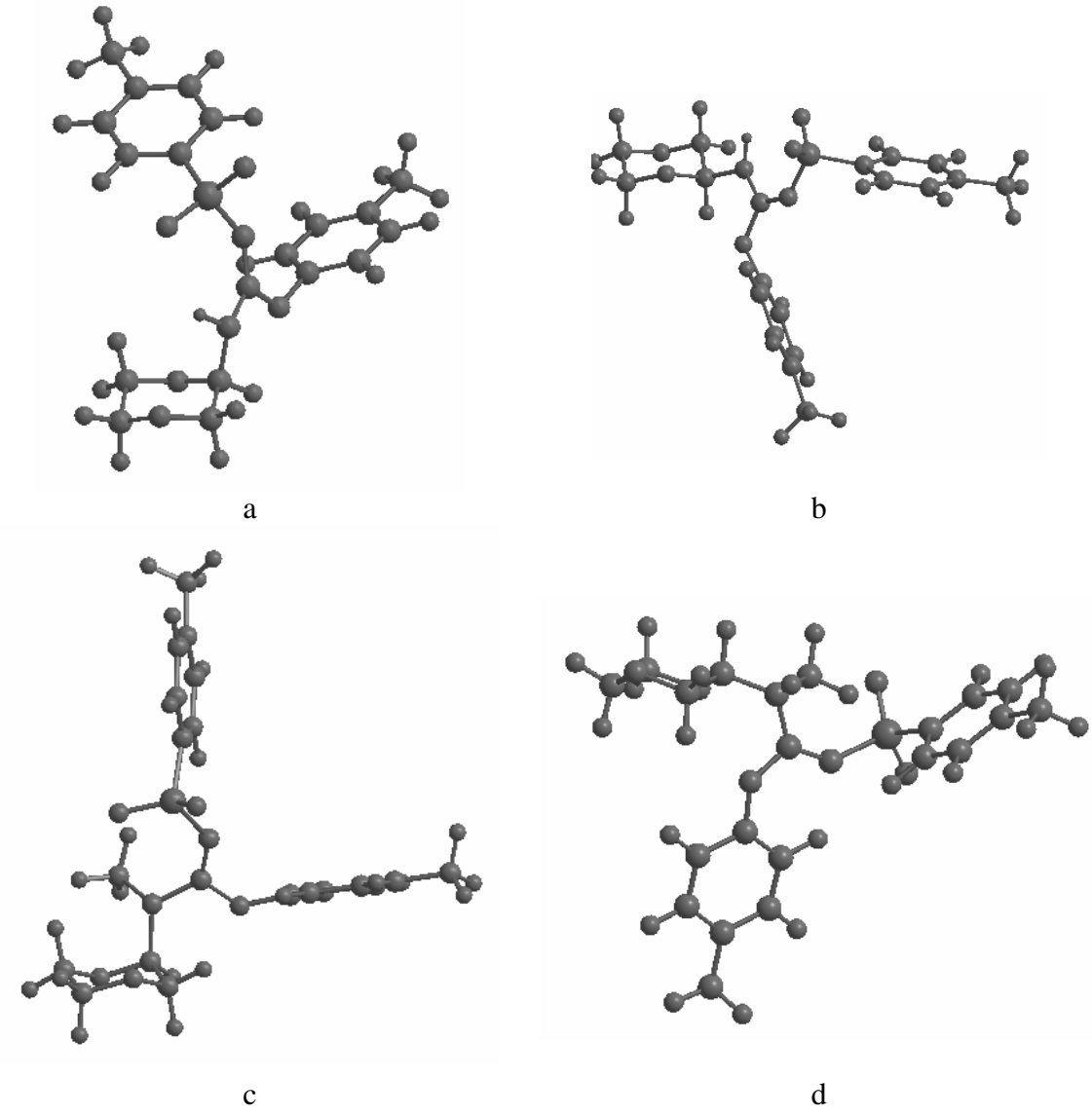

Fig. 2. Molecular structure of (a) **1*E***$_{ax}$, (b) **1*E***$_{eq}$, (c) **1-**NMe***E***$_{ax}$ and (d) **1-**NMe***E***$_{eq}$ calculated by semiempirical AM1.

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