# $^{13}$C AND $^{15}$N NMR SPECTRA OF 6-SUBSTITUTED BICYCLO[3,3,1]7-NONANONE NITRONATES

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$^{13}$C and $^{15}$N NMR spectra (at the natural abundance level of $^{15}$N) have been measured and interpreted for bicyclic reaction products from 1,3,5-trinitrobenzene and cyclohexanone (*I*), cycloheptanone (*II*), cyclooctanone (*III*), acetone (*IV*), 2,4-pentanedione (*V*), 3-methyl-2,4-pentanedione (*VI*), 1-phenyl-1,3-butanedione (*VII*), and methyl acetoacetate (*VIII*). The compounds *I*—*IV* and *VI* exist in keto form, the compounds *V*, *VII*, and *VIII* are mixtures of keto and enol forms. Slow keto-enol equilibria in hexadeuteriodimethyl sulphoxide solutions of *VII* and *VIII* have been followed by $^{13}$C NMR spectra.

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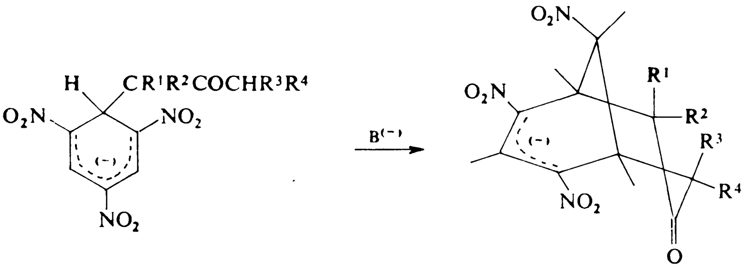
Base catalyzed cyclization of the Meisenheimer complexes derived from 1,3,5-trinitrobenzene and C-acids with two α-carbon atoms carrying protons produces stable compounds (*A*) whose structure was determined by $^{1}$H NMR spectra in many cases[1-5].

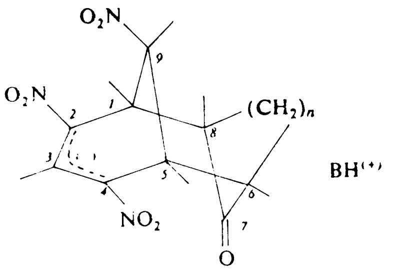


(*A*)

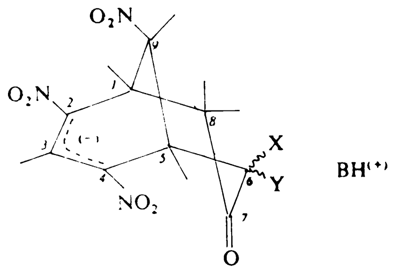
These compounds contain a bicyclo[3,3,1]nonane skeleton and a conjugated dinitropropenide system. They have one or several chiral centres and can exist in form of various isomers and conformers. These complicated structures could only be solved partially by $^{1}$H NMR spectra. The $^{13}$C NMR spectra of the said substances have not been published yet.

## EXPERIMENTAL

Preparation of compounds *I* to *VIII*: 0·63 g (about 3 mmol) 1,3,5-trinitrobenzene was dissolved



*I*



*VIII*

*I*: $n = 3$
*II*: $n = 4$
(+) *III*: $n = 5$
$BH^{(+)} = NH_2(C_2H_5)_2$ for *I*–*IV*
(+)
$BH^{(+)} = NH(C_2H_5)_3$ for *V*–*VIII*

*IV*: $X = Y = H$
*V*: $X = COCH_3$, $Y = H$
*VI*: $X = COCH_3$, $Y = CH_3$
*VII*: $X = COC_6H_5$, $Y = H$
*VIII*: $X = COOCH_3$, $Y = H$

in about 15 mmol of the corresponding C-acid. A few drops of dimethyl sulphoxide were used for the dissolution in the case of 1-phenylbutane-1,3-dione. The solution was treated with 6 mmol diethylamine (for *I* to *IV*) or triethylamine (for *V* to *VIII*). The reaction course was followed by electronic spectra of the solutions prepared by dilution of the reaction mixtures with methanol. After disappearance of absorption bands with $\lambda_{max}$ about 475 and 560 nm (the compounds *I* to *VIII* have a band with $\lambda_{max}$ at 500—510 nm in visible region), the warm reaction mixture was repeatedly extracted with ether, the oily residue was dissolved in warm methanol and filtered with charcoal. The raw product was precipitated with dry ether, collected by suction, and crystallized from a methanol-ether mixture. The compound *II* was purified by chromatography on neutral silica gel with 1 : 1 (by vol.) methanol-dioxane (+0·1% diethylamine) as eluent. We confirmed the structure of compounds *I*, *II*, *IV* and *V* by comparison of their $^1H$ NMR spectra with those given in refs[2–4] (Table I).

For compound *III* ($C_{18}H_{28}N_4O_7$, 412·4) calculated: 52·42% C, 6·84% H, 13·58% N; found: 52·70% C, 6·99% H, 13·76% N.

For compound *VII*, m.p. 148—151°C ($C_{22}H_{28}N_4O_8$, 476·5) calculated: 55·46% C, 5·92% H, 11·76% N; found: 55·72% C, 6·05% H, 11·97% N.

For compound *VIII*, m.p. 162—165°C ($C_{17}H_{26}N_4O_9$, 430·4) calculated: 47·44% C, 6·09% H, 13·02% N; found: 47·24% C, 5·99% H, 12·77% N.

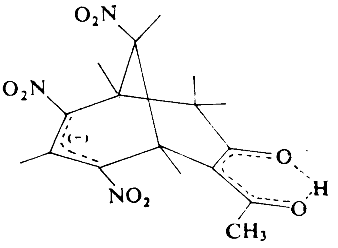
Isolation of compound *VI* in crystalline state failed. Its structure and purity were verified by $^1H$ NMR spectrum.

The $^{13}C$, $^{15}N$, and $^1H$ NMR spectra were measured at 25·047, 10·095, and 99·602 MHz, respectively, using a JEOL JNM-FX 100 apparatus and 10—20% (w/v) solutions of the compounds in hexadeuteriodimethyl sulphoxide. The experimental conditions are given in ref.[6]. The signal of the solvent was used as internal standard ($\delta$ 39·60). The $\delta(^{15}N)$ values are related to external neat nitromethane[7] (25% $^{15}N$), the negative values denote an upfield shift.

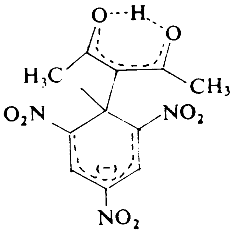
## RESULTS AND DISCUSSION

The number of signals in the $^{13}C$ NMR spectra of compounds *I*–*IV* agreed with the number of magnetically non-equivalent carbon atoms which means that the compounds only exist in the form of a single of possible isomers. The lowest-field signal ($\delta$ 207–216) belongs to carbonyl group. In the region of absorption of $sp^2$ carbon atoms there are two signals. The higher-field signal belongs to quaternary carbon atoms C(2) and C(4) carrying the nitro groups. Assignment of signals of C(1) + + C(5), C(6) + C(8), and C(9) was carried out on the basis of the proton coupled spectra and verified by selective decoupling from $^1$H . Tables I and II give the values of $^1$H and $^{13}$C chemical shifts, respectively. The mutually corresponding carbon atoms in compounds *I–IV* have relatively constant chemical shifts. All the compounds only exist in keto form. Carbon atoms of diethylamonium ion have the chemical shifts $\delta(NCH_2)$ 41·6 ± 0·1 and $\delta(CH_3)$ 11·3 ± 0·1.

The $^{13}$C NMR spectrum of compound *V* is considerably complex. The compound exists, in its solution in hexadeuteriodimethyl sulphoxide and probably also in crystalline state, in the form of a mixture of isomers with predominating enol form *Va*.



*Va*



*IX*

Intensity of the 13 signals of carbon atoms of this enol form is about five times larger than that of the other signals in the spectrum. The quaternary C(6) atom of enol from *Va* has the chemical shift $\delta$ 109·63. A similar value of chemical shift ($\delta$ 112·67) was found[6] for the corresponding carbon atom of enol form of the Meisenheimer complex *IX*. The existence of enol form *Va* is also confirmed by the chemical shifts of carbon atoms of the C=O groups ($\delta$ 183·93 and 196·97). The same groups in enol form of 2,4-pentanedione have the chemical shift $\delta$ 191·01, whereas the carbonyl groups of the keto form[6] have the chemical shift $\delta$ 203·82 (all the data measured in hexadeuteriodimethyl sulphoxide). The carbon atoms of the dinitropropenide system of compound *Va* give 3 signals. The signal with $\delta$ 127·88 was split into a doublet in the proton coupled spectrum and, hence, was assigned to C(3) atom. The signals of the C(1)H, C(5)H, and C(9)H groups were assigned on the basis of selective decoupling: $\delta_C$ 80·43 [C(9)], [$\delta_H$ 5·31 (9-H)]; $\delta_C$ 33·16 [C(5)], [$\delta_H$ 5·18 (5-H)]; $\delta_C$ 31·23 [C(1)], [$\delta_H$ 4·20 (1-H)]. According to the proton-decoupled spectrum, the signal with $\delta$ 37·85 belongs to the methylene group.

Besides the enol form *Va* the mixture also contains the enol form *Vb* (*Va*/*Vb* ~ 10) and the keto form *Vc* (*Va*/*Vc* ~5). The compounds *Vb* and *Vc* were characterized by chemical shifts of carbon atoms of $\mathrm{C{=}O}$ and/or $\mathrm{C} \mathrel{\substack{\cdots \\ -}} \mathrm{O}$ groups (C(6) and C(9)) (Table II). The chemical shifts of these carbon atoms of the enol forms *Va* and *Vb* are very close to each other. In the absorption region of $sp^2$ carbon atoms we found 5 out of 6 signals of the compounds *Vb* and *Vc* ($\delta$ 129·34, 127·94, 125·72, 122·67, 120·57). However, their assignment is impossible, the same being true of the assignment of the absorptions to the carbon atoms C(1), C(5), C(8), and $CH_3$ ($\delta$ 36·15, 34·74, 34·51, 33·28, 30·77, 28·31, 24·45). The enol forms *Va* and *Vb* differ probably in their arrangement at C(9). Ratio of the tautomers *V* remains constant in hexadeuteriodimethyl sulphoxide solution for several days.

The 6-methyl group in compound *VI* prevents formation of the enol form analogous to that of compound *V*. In accordance therewith we could find two signals of carbonyl groups ($\delta$ 205·80 and 207·10). The carbonyl groups of the starting substance[6] have the chemical shift $\delta$ 206·04. The signal with $\delta$ 68·07 corresponds to the quaternary C(6) atom. Selective decoupling enables assignment of the absorptions of the C(1) and C(5) atoms: $\delta_C$ 35·20 [C(1)], [$\delta_H$ 4·24 (1-H)]; $\delta_C$ 41·01 [C(5)], [$\delta_H$ 5·00 (5-H)]. The com-

TABLE I

Selected $^1H$ chemical shifts (ppm) for 6-substituted bicyclo[3,3,1]7-nonanone nitronates in hexadeuteriodimethyl sulphoxide at 25°C

| Compound | Tautomer | 1-H | 5-H | 3-H | 9-H | Reference |
|---|---|---|---|---|---|---|
| *I* | ketone | 4·30 | | 8·21 | 6·30 | 4 |
| *II* | ketone | 4·02 | | 8·18 | 5·80 | 4 |
| *III* | ketone | 4·09 | | 8·22 | 6·25 | *d* |
| *IV* | ketone | 4·52 | | 8·51 | 5·72 | 2 |
| *Va* | enol | 4·20 | 5·18 | 8·40 | 5·31 | 3 |
| *VI*[a] | ketone *a* | 4·24 | 5·00 | 8·34 | 5·56 | *d* |
| | ketone *b* | 4·32 | 4·55 | 8·34 | 5·80 | *d* |
| *VII*[b] | ketone | 4·41 | 4·65 | 8·36 | 5·81 | *d* |
| *VIII*[c] | enol *a* | 4·12 or | 4·14 | 8·22 | 5·22 | *d* |
| | ketone *b* | 4·33 | 4·72 | 8·29 | 5·79 | *d* |
| | ketone *c* | 4·33 | 4·72 | 8·33 | 5·79 | *d* |

[a] $\delta(CH_3)$ 1·10 and 0·93 for *VIa* and *VIb*, respectively; $\delta(COCH_3)$ 2·20. [b] $\delta$(*ortho*) 8·27; $\delta$(*meta,para*) 7·84–7·54; $\delta$(COCHCO) 4·65. [c] $\delta(OCH_3)$ 3·74, 3·78, and 3·55 for *VIIIa*, *VIIIb*, and *VIIIc*, respectively; $\delta$(OH) 12·2 for *VIIIa*. [d] This communication.

TABLE II

$^{13}C$ chemical shifts of carbons in 6-substituted bicyclo[3,3,1]7-nonanone nitronates in hexadeuteriodimethyl sulphoxide at 25°C

| Compound | C(1)/C(5) | C(2) or C(4) | C(3) | C(6) | C(7) | C(8) | C(9) | |
|---|---|---|---|---|---|---|---|---|
| *I* | 42·41 | 122·85 | 127·82 | 48·84 | 215·64 | 48·84 | 77·92 | $\delta(CH_2)$ 30·53 (2 C); 18·89 (1 C) |
| *II* | 41·01 | 123·32 | 127·55 | 53·16 | 210·97 | 53·16 | 78·23 | $\delta(CH_2)$ 28·21; 26·39 |
| *III* | 40·13 | 122·85 | 127·31 | 53·92 | 214·50 | 53·92 | 76·70 | $\delta(CH_2)$ 28·97 (2 C); 23·87 (2 C); 30·85 (1 C) |
| *IV* | 35·10 | 123·26 | 128·00 | 44·34 | 207·91 | 44·34 | 81·25 | |
| *Va* | 31·23;<br>33·16 | 124·08;<br>125·19 | 127·88 | 109·63 | 183·93<br>or 196·97 | 37·85 | 80·43 | $\delta(CO)$ as C(7); $\delta(CH_3)$ 25·21 |
| *Vb* | [a] | [a] | [a] | 108·75 | 183·57<br>or 196·62 | [a] | 79·15 | $\delta(CO)$ as C(7) |
| *Vc* | [a] | [a] | [a] | 67·74 | 201·89<br>or 204·05 | [a] | 81·66 | $\delta(CO)$ as C(7) |
| *VIa* | 35·20;<br>41·01 | 118·92;<br>122·26 | 128·25 | 68·07 | 205·80<br>or 207·10 | 42·95 | 79·58 | $\delta(CO)$ as C(7); $\delta(CO\underline{C}H_3)$ 25·39; $\delta(C\underline{C}H_3)$ 17·41 |
| *VIb* | 35·74;<br>40·59 | 121·39;<br>122·56 | 128·17 | 68·07 | 207·10<br>or 209·37 | 48·26 | 81·49 | $\delta(CO)$ as C(7); $\delta(CO\underline{C}H_3)$ 25·40; $\delta(C\underline{C}H_3)$ 11·93 |
| *VIIa* | 34·37;<br>38·13 | 120·15;<br>123·32 | 127·90 | 61·97 | 194·99<br>or 204·34 | 45·24 | 78·99 | $\delta(CO)$ as C(7); $\delta(i)$ 134·71; $\delta(o)$ 129·25<br>$\delta(m)$ 129·13; $\delta(p)$ 134·59 |
| *VIIb* | | | | 109·29 | 172·98[b] | | 78·00 | |
| *VIIIa* | 31·29 or<br>31·59 | 123·08;<br>124·90 | 127·94 | 99·51 | 171·06<br>or 171·35 | 35·27 | 80·03 | $\delta(CO)$ as C(7); $\delta(OCH_3)$ 52·06 |
| *VIIIb* | | | | | 202·71 | | 80·26 | $\delta(CO)$ 167·25; $\delta(OCH_3)$ 58·55 |
| *VIIIc* | | 122·85<br>119·87 | 128·29 | | 202·47 | | 79·26 | $\delta(CO)$ 167·60; $\delta(OCH_3)$ 53·35 |

[a] See the text; [b] $\delta(\gt C{-}OH)$.

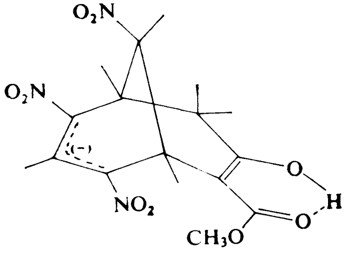
pound isolated immediately after finishing the cyclization reaction of 1,3,5-trinitrobenzene with 3-methyl-2,4-pentanedione is only formed by one isomer *VIa*. After about three months standing of compound *VIa* with excess triethylamine its $^1$H NMR spectrum showed formation of further signals which were assigned to the *VIb* isomer which differs from *VIa* probably in the arrangement at the chiral centre C(6). No such isomerization takes place in solution of the compound *VIa* in hexadeuteriodimethyl sulphoxide. Table II gives the chemical shifts of carbon atoms of compounds *VIa* and *VIb*.

The $^{13}$C NMR spectrum of compound *VII* measured immediately after dissolution in hexadeuteriodimethyl sulphoxide shows 16 signals which agrees with the number of non-equivalent carbon atoms in the molecule. We found 2 signals of carbonyl groups ($\delta$ 194·99 and 204·34) and one signal of C(6) atom with $\delta$ 61·97 (a doublet in the proton coupled spectrum). This means that the compound *VII* exists only in keto form immediately after dissolution and probably also in crystalline state. The signals of C(3) atom and *ortho* carbon atoms of phenyl group were assigned on the basis of selective decoupling: $\delta_{\mathrm{H}}$ 8·36 [(3-H)], $\delta_{\mathrm{ortho}}$ 8·27. For the protons 1-H, 5-H and 6-H the $^1$H NMR spectrum shows two broadened signals with chemical shifts 4·41 and 4·65 and relative intensities 1 : 2. The signal with $\delta$ 4·41 was assigned to the 1-H proton. According to the selective decoupling, the C(1) atom was assigned the signal with $\delta_{\mathrm{C}}$ 34·37. The 5-H and 6-H protons are then connected with the signals of the C(5) and C(6) carbon atoms with $\delta$ 38·13 and 61·97, respectively.

A repeated measurement carried out about 2 h after dissolution of the compound in hexadeuteriomethyl sulphoxide showed further signals in the $^{13}$C NMR spectrum which were assigned to the carbon atoms of the enol form *VIIb* being formed. Out of them the most characteristic are the signals with $\delta$ 172·98 ($>$C—OH) and 109·29 (the quaternary C(6)). The chemical shifts of these signals are very close to those in the spectra of enol forms of the compounds *V* and *VIII*. The signal with $\delta$ 78·00 belongs to the carbon atom C(9) of the enol form *VIIb* formed. The other carbon atoms of the enol form *VIIb* either have chemical shifts very close to those of the carbon atoms of the keto form or could not be assigned (mainly in the aromatic region) due to the enol form being formed in an amount of only 15–20% (according to the $^{13}$C NMR spectrum). The described changes in the $^{13}$C NMR spectrum were quite reproducible and repeated in several independently prepared samples. The time change in the $^{13}$C NMR spectra prove unambiguously that keto–enol equilibria are established which could not be detected by $^1$H NMR spectra alone.

The $^{13}$C NMR spectrum of compound *VIII* is complex and changes with time. The spectrum measured immediately after dissolution of the compound in hexadeuteriodimethyl sulphoxide only contains 13 signals. The presence of the signals with $\delta$ 171·06 and 171·35 ($COOCH_3$ and/or $>$C—OH) and $\delta$ 99·51 (quaternary C(6)) indicates unambiguously that the compound exists in enol form *VIIIa*. After about 30 min the spectrum already contains considerably intensive signals of two keto forms *VIIIb* and *VIIIc*. After about 30 min of the reaction the compounds are present at the ratio *VIIIa* : *VIIIb* : *VIIIc* = 2 : 1 : 1. The formed keto forms are characterized by the chemical shifts of the carbonyl groups (δ 202·71 and 202·47, respectively) and the ester groups (δ 167·25 and 167·60, respectively). A detailed assignment of all the signals was impossible due to considerable complexity of the spectrum.

During about 24 h another reaction took place after which the compound ratio charged to *VIIIa* : *VIIIb* : *VIIIc* ≈ 2 : 1 : 4. This ratio did not furter change during several days. The described changes were quite reproducible. The time changes in the $^{13}$C NMR spectra can be interpreted in the following way: In crystalline state the compound *VIII* exists probably in the form of the enol *VIIIa*. After dissolution in dimethyl sulphoxide an acid–base-catalyzed reaction takes place, and the keto forms *VIIIb* and *VIIIc* are formed at roughly the same rate under the conditions of kinetic control. In a next phase the following thermodynamically controlled reaction takes place: *VIII* ⇄ *VIIIa* ⇄ *VIIIc*.



*VIIIa*

The keto forms *VIIIb* and *VIIIc* can differ either in configuration at C(9) or in conformation of C(7) = 0 cabonyl group. From comparison of δ($^{13}$C) and δ($^{1}$H) of the compounds *I* – *VII* it is presumed, however, that *VIIIb* and *VIIIc* are diastereoisomers differing by configuration at C(6).

The described reactions whose existence was deduced from $^{13}$C NMR spectra can also be confirmed by $^{1}$H NMR spectra. In the $^{1}$H NMR spectra we observed, in dependence on time, three signals of the 3-H protons and three signals of the ester methoxy groups (Table I) whose intensities changed with time in accordance with the changes in the $^{13}$C NMR spectra.

The content of enol form increases after transformation of 2,4-pentanedione to the Meisenheimer complex *IX* or compound *V*. 2,4-Pentanedione contains about 50% of enol form, the compound *V* contains about 75%, and the complex *IX* only exist in enol form[6]. Similar is the situation of methyl acetoacetate: the keto ester itself contains no enol form detectable by NMR, the Meisenheimer complex with 1,3,5-trinitrobenzene[8] contains about 10%, and compound *VIII* contains about 30% enol form at the equilibrium (all the data measured in hexadeuteriodimethyl sulphoxide). The reverse situation applies to 1-phenyl-1,3-butanedione. The starting diketone is completely enolized in dimethyl sulphoxide, compound *VIII* only contains about 15−20% enol form at the equilibrium*. We suppose that the low enol form content in compound *VII* is due to increased steric requirements of the $C_6H_5-\overset{|}{C}=O$ group when assuming the plane arrangement in the enol form.

The chemical shifts of carbon atoms of triethylammonium ion in compounds *V* to *VIII* are $\delta(NCH_2)$ 46·0 ± 0·1 and $\delta(CH_3)$ 8·7 ± 0·1.

Complete stereochemistry of bicyclo[3,3,1]7-nonanone nitronates has not been solved yet. Strauss[9] presumes configurational stability at the C(9) atom. This stability is also indicated by the constant chemical shifts of the carbon atoms C(2), C(3), and C(4) in compounds *I*−*VIII* (Table II). However, it is not clear whether there is the proton 9-H or nitro group over the dinitropropenide system, since neither the proton 9-H not the nitrogen atom of the nitro group have the chemical shift independent of the rest of the molecule (Tables I and III). Therefore, we tried to dedetermine the configuration at the C(9) atom in the enol form *Va* by means of the nuclear Overhauser effect. In all the cases, however, the irradiation of the proton 3-H did not change the intensity ratio of the proton absorptions 9-H, 5-H, 1-H (1 : 1 : 1). In spite of the orientation of the protons 3-H and 9-H being not optimum for operation of the nuclear Overhauser effect, even in the case that the proton 9-H

TABLE III

$^{15}N$ chemical shifts (ppm) of nitrogen atoms of nitro groups[a] of 6-substituted bicyclo[3,3,1]-7-nonanone nitronates in hexadeuteriodimethyl sulphoxide at 25°C measured in the presence of chromium(III) tris-acetylacetonate (c. 10 mg $ml^{-1}$) and referred to external neat nitromethane

| Compound | 2-N or 4-N | | 9-N |
|---|---|---|---|
| *I* | −16·4 | | +6·2 |
| *IV* | −16·1 | | +6·9 |
| *Va* | −15·3 | −17·7 | +6·7 |
| *VIa* | −13·4 | −16·0 | +5·8 |
| *VII* | −14·7 | −17·3 | +4·8 |

[a] Negative values denote the upfield shift.

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* The NMR spectra of the Meisenheimer complex of 1-phenyl-1,3-butanedione with 1,3,5-trinitrobenzene have not been measured yet.

were over the dinitropropenide system, the negative result of the experiment supports the presumption that there is the nitro group over the dinitropropenide system.

1,3,5-Trinitrobenzene contains 3 isochronous nitro groups with the chemical shift $\delta(^{15}N)$ $-18{\cdot}7$ (ref.[6]). Transformation of 1,3,5-trinitrobenzene into compounds *I* – *VIII* changes character of one of the nitro groups from Ar—$NO_2$ to R—$NO_2$, which is connected with a change in $\delta(^{15}N)$ to a value of $+4{\cdot}8$ to $+6{\cdot}9$ (Table III). These values correspond roughly to $\delta(^{15}N)$ of aliphatic nitro compounds[10]. If the produced bicyclic nitronates possess a plane of symmetry (compounds *I* and *II*), then the remaining nitro groups are isochronous. In other cases the two nitro groups have different chemical shifts $\delta(^{15}N)$. The $\delta(^{15})N$ chemical shifts of nitrogen atoms of the nitro groups bound to the conjugated propenide system are within the interval from $-13{\cdot}4$ to $-17{\cdot}7$ and agree with those of nitro groups at 4 position of the cyclohexadienide system of the Meisenheimer complexes from 1,3,5-trinitrobenzene and carbanions[6].

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