PREPARATION OF 3-CYANO-4-(R-VINYL)-5,5-DIMETHYL-Δ^3-BUTENOLIDES AND SUBSTITUENT EFFECTS ON THEIR INFRARED SPECTRA

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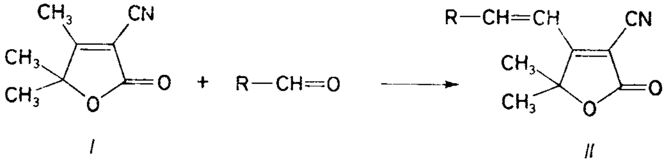
Twenty nine 3-cyano-4-(R-vinyl)-5,5-dimethyl-Δ^3-butenolides were prepared by condensation of 3-cyano-4,5,5-trimethyl-Δ^3-butenolide with aliphatic and aromatic aldehydes. The wave numbers of C=O, C≡N and C=C stretching vibrations of synthesized compounds were measured in trichloro- and tetrachloromethane. The spectral data were correlated with substituent conotants using Hammet–Brown and the improved and extended Seth-Paul–Van Duyse equation. The statistical results of correlations were compared with those of ethyl α-cyanocinnamates, ethyl benzoates and benzonitriles. The transmission factors for one and two C=C bonds were determined in the regions of $\pi' = 0{\cdot}46-0{\cdot}57$ and $0{\cdot}27-0{\cdot}37$, respectively. In 3-cyano-4-(arylvinyl)-5,5-dimethyl-Δ^3-butenolides a considerable conjugation exists between the arylvinyl moiety and both the C=O and C≡N groups. On the other hand, in 3-cyano-4-(alkylvinyl)-5,5-dimethyl-Δ^3-butenolides probably only the endocyclic C=C bond takes part in a marked resonance interaction with alkyvinyl group. A generally valid relationship was found between the slopes of ν(C=O) vs σ^+ and ν(C≡N) vs σ^+ correlations for different types of compounds measured in $CHCl_3$ and CCl_4.

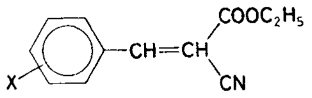
A number of compounds containing unsaturated γ-lactone ring posses a wide range of valuable properties[1-3]. The biological activity of γ-lactones is partially dependent on the properties of ring substituents. It is known, that biologically active compounds, like penicillic acid, vulpenic acid, protoanemonin and others contain in theire structure substituents with C=C bonds attached to the unsaturated γ-lactone ring.



SCHEME 1

Following this idea a series of 3-cyano-4-(R-vinyl)-5,5-dimethyl-Δ^3-butenolides (*II*) were synthesized by condensation of 3-cyano-4,5,5-trimethyl-Δ^3-butenolide (*I*) with aliphatic and aromatic aldehydes (Scheme 1).

To investigate the transmission of substituent effects in unsaturated γ-lactone ring the IR spectra of compounds *II* were measured and compared with those of ethyl α-cyanocinnamates (*III*), ethyl benzoates (*IV*) and benzonitriles (*V*).



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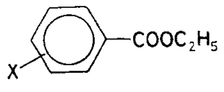

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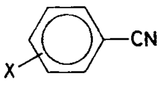

V

EXPERIMENTAL

Melting points were determined on a Kofler block and are uncorrected. IR spectra in the 1 800 to 1 500 cm^{-1} and 2 300—2 100 cm^{-1} regions were taken on a Specord 75 IR (Zeiss, Jena) spectrometer in trichloro- and tetrachloromethane at room temperature using NaCl cell of 0·1, 1·0, 5·0, 10·0, and 20·0 mm thickness. The concentration of the measured solutions were chosen to give transmission between 30—25%. Peak positions were determined by averaging the results of three measurements and are believed to be accurate to ±0·5 cm^{-1}. The instrument calibration was verified by using water vapor and indene stadard spectra.

3-Cyano-4-(R-vinyl)-5,5-dimethyl-Δ^3-butenolides (*II*)

To a solution of NaOH (0·05 g) in absolute methanol (15 ml) 3-cyano-4,5,5-trimethyl-Δ^3-butenolide (*I*, ref.[4]; 0·02 mol) and the corresponding aldehyde (0·022 mol) were added. The mixture after refluxing for 4 h was acidified with dilute (1 : 1) hydrochloric acid. The separated solid was filtered off, washed with water and recrystallized from ethanol using active charcoal.

RESULTS AND DISCUSSION

The electron-withdrawing effects of both the C=O and C=N groups attached to the C=C bond of unsaturated γ-lactone ring are responsible for the strong C—H acidity of 4-methyl group in *I*. Consequently 3-cyano-4,5,5-trimethyl-Δ^3-butenolide (*I*) in alkaline medium undergo condensation with aliphatic and aromatic aldehydes (Scheme 1) affording 3-cyano-4-(R-vinyl)-5,5-dimethyl-Δ^3-butenolides (*II*) in relatively high yields (Table I). On the other hand, if the CN group is lacking in *I*, by much lower yields of products were obtained by condensation with aldehydes[5].

The IR absorption bands of 3-cyano-4,5,5-trimethyl-Δ^3-butenolide (*I*) can be assigned for ν(C=C): 1 656·0 cm^{-1} ($CHCl_3$), 1 657·5 cm^{-1} (CCl_4); for ν(C=O); 1 777·0 cm^{-1} ($CHCl_3$), 1 792·5 cm^{-1} (CCl_4); for ν(C≡N): 2 245·5 cm^{-1} ($CHCl_3$), 2 246·0 cm^{-1} (CCl_4). The IR spectral data for the series of 3-cyano-4-(R-vinyl)-5,5-dimethyl-Δ^3-butenolides (*II*) measured in dilute solutions in $CHCl_3$ and CCl_4 to-

TABLE I

3-Cyano-4-(R-vinyl)-5,5-dimethyl-Δ^3-butenolides of type *II*

No	R	Formula (M.w.)	Calculated/Found			M.p., °C (Yield, %)
			% C	% H	% N	
1	C_6H_5	$C_{15}H_{13}NO_2$ (293·3)	75·30 75·74	5·47 5·81	5·86 6·04	176—178 (88)
2	2-MeC_6H_4	$C_{16}H_{15}NO_2$ (253·3)	75·87 76·11	5·97 6·22	5·53 5·51	130—132 (69)
3	2-$MeOC_6H_4$	$C_{16}H_{15}NO_3$ (269·3)	71·36 71·58	5·61 5·72	5·20 5·01	139—141 (78)
4	2-HOC_6H_4	$C_{15}H_{13}NO_3$ (255·3)	70·58 70·49	5·13 4·95	5·49 5·72	205—206 (81)
5	2-ClC_6H_4[a]	$C_{15}H_{12}ClNO_2$ (273·7)	65·82 65·98	4·42 4·13	5·12 5·30	154—156 (59)
6	4-$NO_2C_6H_4$	$C_{15}H_{12}N_2O_4$ (284·3)	63·38 63·25	4·26 4·07	9·85 10·22	192—193 (78)
7	3-MeC_6H_4	$C_{16}H_{15}NO_2$ (253·3)	75·87 76·02	5·97 6·32	5·53 5·67	160—162 (74)
8	3-$MeOC_6H_4$	$C_{16}H_{15}NO_3$ (269·3)	71·36 71·67	5·61 5·45	5·20 5·13	203—204 (84)
9	3-HOC_6H_4	$C_{15}H_{13}NO_3$ (255·3)	70·58 70·21	5·13 5·29	5·49 5·17	241—241 (77)
10	3-ClC_6H_4[b]	$C_{15}H_{12}ClNO_2$ (273·7)	65·82 65·49	4·42 5·02	5·12 5·50	208—209 (77)
11	3-$NO_2C_6H_4$	$C_{15}H_{12}N_2O_4$ (284·3)	63·38 63·47	4·26 4·91	9·85 9·68	258—259 (81)
12	4-MeC_6H_4	$C_{16}H_{15}NO_2$ (253·3)	75·87 75·69	5·97 6·03	5·53 5·40	151—153 (79)
13	4-$MeOC_6H_4$	$C_{16}H_{15}NO_3$ (269·3)	71·36 71·17	5·61 5·47	5·20 5·61	156—157 (93)
14	4-HOC_6H_4	$C_{15}H_{13}NO_3$ (255·3)	70·58 70·45	5·13 5·50	5·49 5·13	295—296 (64)
15	4-ClC_6H_4[c]	$C_{15}H_{12}ClNO_2$ (273·7)	65·82 66·11	4·42 4·11	5·12 5·01	150—151 (78)
16	4-$NO_2C_6H_4$	$C_{15}H_{12}N_2O_4$ (284·3)	63·28 63·72	4·26 4·30	9·85 10·17	255—256 (84)
17	4-$MeCONHC_6H_4$	$C_{17}H_{16}N_2O$ (296·3)	68·71 69·20	5·44 5·58	9·45 9·32	280—282 (72)

TABLE I
(*Continued*)

No	R	Formula (M.w.)	Calculated/Found			M.p., °C (Yield, %)
			% C	% H	% N	
18	3,4-$(MeO)_2C_6H_3$	$C_{17}H_{17}NO_4$	68·22	5·73	4·68	203—204
		(299·3)	68·40	5·99	4·81	(71)
19	3-MeO-4-HOC_6H_3	$C_{16}H_{15}NO_4$	67·36	5·30	4·91	255—256
		(285·3)	67·70	5·49	4·69	(78)
20	—[d]	—	—	—	—	—
		—	—	—	—	—
21	Fc[e]	$C_{19}H_{17}FeNO_2$	65·70	4·93	4·03	177—178
		(347·3)	65·82	4·72	4·17	(63)
22	4-$Et_2NC_6H_4$	$C_{19}H_{22}N_2O_2$	73·52	7·15	9·02	121—122
		(310 : 4)	73·59	7·40	8·87	(66)
23	4-$Me_2NC_6H_4$	$C_{17}H_{18}N_2O$	72·32	6·42	9·92	134—135
		(282·3)	72·08	6·64	10·11	(67)
24	Me	$C_{10}H_{11}NO_2$	67·78	6·26	7·91	215—216
		(177·2)	67·42	6·41	8·13	(71)
25	Et	$C_{11}H_{13}NO_2$	69·09	6·85	7·33	180—181
		(191·23)	68·81	6·49	7·21	(69)
26	Pr	$C_{12}H_{15}NO_2$	70·22	7·37	6·83	156—157
		(205·3)	70·45	7·09	6·59	(74)
27	iPr	$C_{12}H_{15}NO_2$	70·22	7·37	6·83	211—212
		(205·3)	70·03	7·53	6·97	(75)
28	3,4-$CH_2O_2C_6H_3$	$C_{16}H_{13}NO_4$	67·84	4·62	4·95	215—216
		(283·3)	67·58	4·38	5·12	(88)
29	2-Thi[f]	$C_{13}H_{11}NO_2S$	63·65	4·32	5·71	153—154
		(245·3)	63·90	4·49	5·90	(86)

[a] Calculated 12·95% Cl, found 13·19% Cl; [b] calculated 12·95% Cl, found 12·69% Cl; [c] calculated 12·95% Cl, found 12·80% Cl; [d] the preparation of compound will be published elsewhere; [e] ferrocenyl, calculated 16·12% Fe, found 16·35% Fe; [f] 2-thienyl, calculated 13·07% S, found 13·21% S.

gether with corresponding $\sigma^+(X)$ and $X^+(R)$ substituent constants are given in Table II.

In compounds *II* (R = alkyl) the wave numbers of $\nu(C{=}C)$ are shifted by ~14 cm^{-1} to lower values (comparing to *I*). The wave numbers of C≡N stretching vibration exhibit only a small decrease (2·5 – 3·0 cm^{-1}), while the values of νC=O) wave numbers remain practically the same as in the case of starting compound (*I*). On the other hand, in compounds *II* (R = aryl of heteroaryl) the wave numbers of all ν(C=C), ν(C=O), and ν(C≡N) vibrations are significantly shifted to lower

TABLE II

IR spectral data (in cm^{-1}) and substituent constants for 3-cyano-4-(R-vinyl)-5,5-dimethyl-Δ^3-butenolides of type *II*

No.	CHCl$_3$			CCl$_4$		σ^+(X)[a]	X^+(R)[b]
	ν(C=O)	ν(C≡N)	ν(C=C)	ν(C=O)	ν(C≡N)		
1	1 767·5	2 235·5	1 621·0	1 782·0	2 233·5	0·00	1·070
2	1 766·5	2 234·5	1 620·5	1 780·5	2 232·0	−0·31[c]	0·960
3	1 764·5	2 234·5	1 613·5	1 780·0	2 234·0	−0·78	0·88
4	1 763·0	2 235·0	1 613·0	—[d]	—[d]	−0·92[c]	0·858[c]
5	1 770·0	2 236·0	1 621·0	1 783·0	2 235·0	0·11[c]	1·235
6	1 771·5	2 240·0	1 626·0	1 784·0	2 238·0	0·79[c]	1·470
7	1 766·5	2 236·0	1 622·0	1 780·0	2 235·0	−0·07	1·060
8	1 766·5	2 236·0	1 623·0	1 781·5	2 235·0	0·05	1·089
9	1 766·5	2 237·0	1 625·0	—[d]	—[d]	−0·04	1·067[e]
10	1 768·5	2 237·0	1 624·0	1 784·0	2 234·5	0·40	1·172
11	1 770·0	2 238·0	1 628·5	—[d]	—[d]	0·67	1·236
12	1 765·5	2 234·5	1 624·0	1 780·0	2 233·0	−0·31	1·003
13	1 763·0	2 233·5	1 613·5	1 778·5	2 232·0	−0·78	0·891
14	1 765·0	2 235·0	1 603·0	—[d]	—[d]	−0·92	0·858
15	1 768·5	2 236·0	1 622·5	1 783·0	2 234·0	0·11	1·103
16	1 771·5	2 237·5	1 626·5	1 785·5	—[d]	0·79	1 265
17	1 766·0	2 234·5	1 622·5	—[d]	—[d]	−0·60	0·934
18	1 763·5	2 234·0	1 620·0	1 779·5	2 232·0	−0·73	0·890
19	1 763·5	2 234·0	1 623·0	—[d]	—[d]	−0·83	0·870
20	1 763·5	2 234·0	1 617·0	1 779·0	2 233·5	−0·94	0·853
21	1 760·5	2 233·0	1 603·5	1 778·0	2 231·5	−1·55	0·708
22	1 755·0	2 229·5	1 610·0	1 774·5	2 229·0	−2·09	0·582[e]
23	1 756·0	2 229·5	1 612·0	1 775·0	2 229·5	−1·70	0·672
24	1 778·5	2 243·5	1 643·5	—[d]	—[d]	—	1·807
25	1 778·0	2 243·5	1 643·0	—[d]	—[d]	—	1·758
26	1 776·5	2 243·0	1 642·0	—[d]	2 240·5	—	1·751
27	1 778·0	2 243·0	1 642·0	—[d]	—[d]	—	1·708
28	1 764·5	2 234·5	1 616·0	1 780·0	2 232·5	—	—
29	1 764·5	2 235·0	1 607·5	1 781·0	2 231·0	−1·66	0·682

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[a] Refs[6,7]; [b] refs[6,8–10]; [c] the σ^+(X) or X^+(R) values for *para*-position were used; [d] insoluble; [e] calculated according to equation $X^+(R) = 0{\cdot}238\sigma^+(X) + 1{\cdot}077$ (ref.[6]).

values. This indicates that in 3-cyano-4-(arylvinyl)-5,5-dimethyl-Δ^3-butenolides a good conjugation exist between all multiple bonds in the whole molecule, while in 4-alkylvinyl derivatives probably only the endocyclic C=C bond takes part in a significant resonance interaction with alkylvinyl moiety.

It is evident from Table II, that the ν(C=O) bands are sensitive to the effect of solvent, while the positions of ν(C≡N) bands remain practically unaffected. That means that in compounds *II* the C=O group preferably forms intermolecular hydrogen bond with trichloromethane, however, the cyano group does not interact with hydrogen atom of $CHCl_3$.

To investigate the transmission of substituent effects in unsaturated γ-lactone system the wave numbers of ν(C=O) and ν(C≡N) for series of compounds *II* were correlated with substituent constants. In correlations Brown–Okamoto's $\sigma^+(X)$ constants were used for aromatic or heteroaromatic compounds. Since the system is well conjugated, the use of Hammett $\sigma(X)$ values in correlations exhibit statistically less significant results. To include also 4-alkylvinyl derivatives into the correlations $X^+(R)$ constants of the improved and extended Seth-Paul–Van Duyse equation[6] were employed. The statistical results of correlation analysis are summarized in Table III. The data of some compounds were excluded from correlations (as indicated in Table III). It was mostly the case of 4-dimethylaminophenyl and 4-diethylaminophenyl derivatives (22 and 23), in which the strong resonance interaction between the nitrogen atom lone-pair electrons and both the C=O and C≡N groups

TABLE III

Statistical results of correlation analysis for series of compounds *II*

Correlation	Solvent	n^a	r^b	ϱ^c	q^d	s^e
$\nu(C=O)$ vs $\sigma^+(X)^f$	$CHCl_3$	21	0·956	4·42 ± 0·31	1 767·5 ± 0·2	0·89
$\nu(C=O)$ vs $\sigma^+(X)^g$	CCl_4	17	0·964	3·55 ± 0·25	1 782·0 ± 0·2	0·84
$\nu(C\equiv N)$ vs $\sigma^+(X)^h$	$CHCl_3$	16	0·928	2·24 ± 0·24	2 235·9 ± 0·1	0·51
$\nu(C\equiv N)$ vs $\sigma^+(X)^i$	CCl_4	10	0·936	2·21 ± 0·29	2 233·8 ± 0·3	0·74
$\nu(C=O)$ vs $X^+(R)^h$	$CHCl_3$	25	0·982	15·40 ± 0·61	1 750·6 ± 0·7	0·97
$\nu(C=O)$ vs $X^+(R)^k$	CCl_4	17	0·983	12·69 ± 0·62	1 768·2 ± 0·7	0·82
$\nu(C\equiv N)$ vs $X^+(R)^l$	$CHCl_3$	24	0·987	10·12 ± 0·35	2 225·3 ± 0·4	0·55
$\nu(C\equiv N)$ vs $X^+(R)^m$	CCl_4	14	0·992	9·72 ± 0·35	2 223·3 ± 0·4	0·47

[a] Number of compounds used in correlation; [b] correlation coefficient; [c] slope; [d] intercept; [e] standard deviation; [f] compounds 22, 23, and 29 were omitted; [g] compounds 4, 14, 19, and 29 were omitted; [h] compounds 4, 6, 9, 14, 21, 22, 23, and 29 were omitted; [i] compounds 3, 6, 7, 8, 20, 21, and 29 were omitted; [j] compounds 22, 23, and 29 were omitted; [k] compounds 6, 22, 23, and 29 are omitted; [l] compounds 5, 22, 23, and 29 were omitted; [m] compounds 3, 7, 8, 20, and 29 were omitted.

causes an anomalous decrease in the $\nu(C{=}O)$ and $\nu(C{\equiv}N)$ values. The 2-thienyl derivative (*29*) was omitted in all cases since the high negative $\sigma^+(X)$ or low $X^+(R)$ value (found in literature) seem to be enhanced. In some other cases compounds containing *ortho*- substituents or a hydroxy group were also rejected because of unaccurate value of substituent constant or due to hydrogen bonding between the OH group and other parts of the molecule.

The data in Table III show that the C=O group is always more sensitive to substituent effects than the C≡N group attached at the same place to the system conjugated with substituted benzene ring. This is in a good agreement with results of Yukhnovskii[11,12], who investigated the $\nu(C{=}O)$ vs $\sigma^+(X)$ and $\nu(C{\equiv}N)$ vs $\sigma^+(X)$ correlations in series of ethyl α-cyanocinnamates (*III*), ethyl benzoates (*IV*) and benzonitriles

TABLE IV

Transmission factors for one and two double bond systems in 3-cyano-4-(R-vinyl)-5,5-dimethyl-Δ^3-butenolides (*II*) calculated by Eqs (*2*)–(*5*)

Correlation	Solvent	$\pi'(C{=}C)$ Eq. (*2*)	$\pi'(C{=}C{-}C{=}C)$	
			Eq. (*3*) or (*4*)	Eq. (*5*)
$\nu(C{=}O)$ vs σ^+	$CHCl_3$	0·56	0·37	0·31
$\nu(C{=}O)$ vs σ^+	CCl_4	0·57	0·37	0·32
$\nu(C{\equiv}N)$ vs σ^+	$CHCl_3$	0·46	0·27	0·21
$\nu(C{\equiv}N)$ vs σ^+	CCl_4	0·54	0·33	0·29

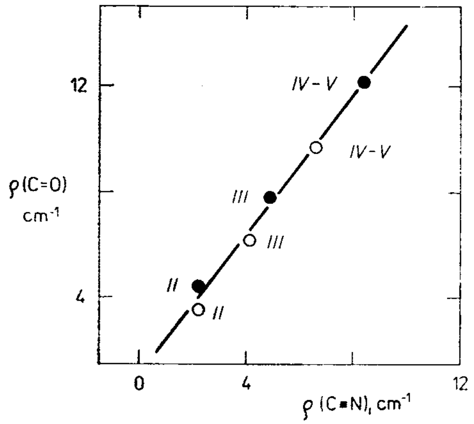

FIG. 1

Plot of $\varrho(C{=}O)$ vs $\varrho(C{\equiv}N)$ for systems with conjugated C=O and C≡N groups attached at the same or analogous place in the molecule; ● values measured in CCl_4, ○ values measured in $CHCl_3$

(*V*). Comparing the slopes $\varrho(C{=}O)$ of $\nu(C{=}O)$ vs $\sigma^+(X)$ correlations with $\varrho(C{\equiv}N)$ i.e. the slopes of $\nu(C{\equiv}N)$ vs $\sigma^+(X)$ correlations for series of compounds *II* (Table III) and *III* – *V* (refs[11,12]) a linear dependence (Eq. (*1*), see Fig. 1) can be found.

$$\varrho(C{=}O) = 1{\cdot}31\varrho(C{\equiv}N) + 1{\cdot}07 \qquad (1)$$

As all data for different types of compounds measured in both sovents follow well the straight line in Fig. 1, we can conclude that Eq. (*1*) has a general validity and can be used to predict $\varrho(C{=}O)$ or $\varrho(C{\equiv}N)$ values in any other conjugated systems, in which the C=O and C≡N group are attached at the same or analogous place.

Using the values of slopes (ϱ) for series of compounds *II* – *V* (Table III and refs[11,12]) the transmission factors $\pi'(C{=}C)$ and $\pi'(C{=}C{-}C{=}C)$ for one or two conjugated doule bonds can be determined by Eqs (*2*)–(*4*),

$$\pi'(C{=}C) = \varrho(II)/\varrho(III) \qquad (2)$$

$$\pi'(C{=}C{-}C{=}C) = \varrho(II)/\varrho(IV) \qquad (3)$$

$$\pi'(C{=}C{-}C{=}C) = \varrho(II)/\varrho(V) \qquad (4)$$

or calculated by approach (Eq. (*5*)) published by Yanovskaya et al.[14]

$$\pi'(C{=}C{-}C{=}C) = \pi'^2(C{=}C)\,. \qquad (5)$$

The results are given in Table IV. The transmission factors for one double bond $\pi'(C{=}C) = 0{\cdot}46 - 0{\cdot}57$ are somewhat lower than the values $\pi'(C{=}C) = 0{\cdot}58$ to $0{\cdot}65$ determined by Yukhnovskii[11,12] using the slopes of similar correlations in series *III* – *V*. This difference can be explained by cyclic structure of compounds *II*. Relatively high values of transmission factors $\pi'(C{=}C{-}C{=}C) = 0{\cdot}27 - 0{\cdot}37$ were obtained for system containing two double bonds comparing to similar results of Yanovskaya et al.[13] ($\pi'(C{=}C{-}C{=}C) = 0{\cdot}25$). This indicates again that all multiple bonds in aromatic and heteroaromatic derivatives of *II* are markedly conjugated. The good agreement between the values of $\pi'(C{=}C{-}C{=}C)$ determined using Eqs (*3*) and (*4*) and those calculated by Eq. (*5*) strongly supports the conclusions of Yanovskaya et al.[13] or the idea of Bowden[14] on exponential weakening of substituent effects in conjugated systems.

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