SYNTHESIS OF 3-(5-ALKYL-4-ACETYL(ETHOXYCARBONYL))-2-PROPENOATES FROM DERIVATIVES OF 5-PHENOXY-2-FURYLMETHYLENE

Štefan MARCHALÍN[a], Vladimír MLYNÁRIK[b], Anna STAŇOVÁ[a] and Dušan ILAVSKÝ[a]
[a] *Department of Organic Chemistry, Slovak Technical University, 812 37 Bratislava*
[b] *Czechoslovak Institute of Metrology, 842 55 Bratislava*

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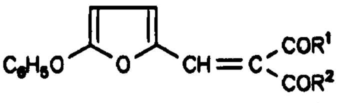
3-(2-Furyl)propenoates *IV* – *VIII* were obtained by treatment of 5-phenoxy-2-furylmethylene derivatives *I* – *III* with alcoholic hydrogen chloride in one step. The probable transition mechanism of synthons *I* – *III* into the 2,4,5-trisubstituted furans is presented. The geometric isomers *E-IV*, *Z-V* and *Z-VI*, isolated in pure form, were transformed into the corresponding acids *E-IX*, *Z-IX* and *E-X* by base-catalyzed hydrolysis. Acid hydrolysis of the 1,3-diketone *I* afforded (4Z,6Z)-6-acetyl-7-hydroxy-2,4,6-octatrien-4-olide (*XI*). Structure of the products was verified by spectral (IR, UV, NMR and mass) methods.

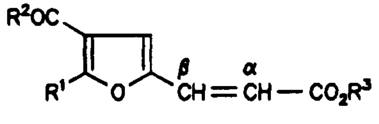
Furan derivatives containing an alkoxy or a trimethylsiloxy group are advantageous protected 2-buten-4-olides used in organic synthesis[1 – 4]. Acid hydrolysis of 2-substituted 5-methoxyfurans leading to 4-alkylidene-2-buten-4-olides was utilized in the chemistry of natural products when synthesizing e.g. digitoxigenin[5] or brassanolide[6]. In continuation of our research on the reactivity of activated derivatives of furylethylene[7 – 9] we turned our attention to acid hydrolysis of the easily accessible 5-phenoxy-2-furylmethylene derivatives *I* – *III*.

As found, 3-(5-phenoxy-2-furylmethylene)-2,4-pentanedione (*I*) furnished with methanolic hydrogen chloride in methanol a product, which crystallized as white crystals, m.p. 106 – 107 °C in good yield (70%). This compound was ascribed the structure of methyl (*E*)-3-(4-acetyl-5-methyl-2-furyl)propenoate (*E-IV*) on the basis of elemental analysis and spectral data. Isomerism of the double bond of the propene residue was characterized unambiguously by the absorption band in the IR spectrum at 972 cm^{-1} and a vicinal coupling constant in the ^1H NMR spectrum $^3J(\alpha,\beta) = 15.9$ Hz. The ^1H NMR analysis of the crude product *IV* proved this material to be a mixture of *E* and *Z* isomers in a 45 : 55 ratio. Similarly, the reaction in ethanol yielded a mixture of *E* and *Z* isomers of the ester *V* in a 40 : 50 ratio; crystallization from ethanol gave the less soluble *Z* isomer in pure state.

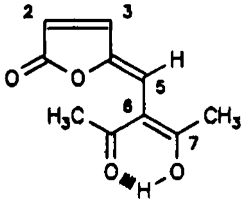
The crude product *V* underwent isomerization into the *E* counterpart in ethanolic hydrogen chloride. The H-3 proton of the *Z-V* isomer resonating at δ 7.97 is considerably deshielded (Δδ 1.17, $^3J(\alpha,\beta)$ 12.8 Hz) with respect to that of *E-V*. The ^{13}C NMR spectra of isomers *V* were very close as far as they can even be identified unequivocally by ^{1}H NMR spectra.



I, $R^1 = R^2 = CH_3$
II, $R^1 = CH_3$; $R^2 = OC_2H_5$
III, $R^1 = CH_2Cl$; $R^2 = OC_2H_5$



IV, $R^1 = R^2 = R^3 = CH_3$
V, $R^1 = R^2 = CH_3$; $R^3 = C_2H_5$
VI, $R^1 = CH_3$; $R^2 = OC_2H_5$; $R^3 = CH_3$
VII, $R^1 = CH_3$; $R^2 = OC_2H_5$; $R^3 = C_2H_5$
VIII, $R^1 = CH_2Cl$; $R^2 = OC_2H_5$; $R^3 = CH_3$
IX, $R^1 = R^2 = CH_3$; $R^3 = H$
X, $R^1 = CH_3$; $R^2 = OH$; $R^3 = H$



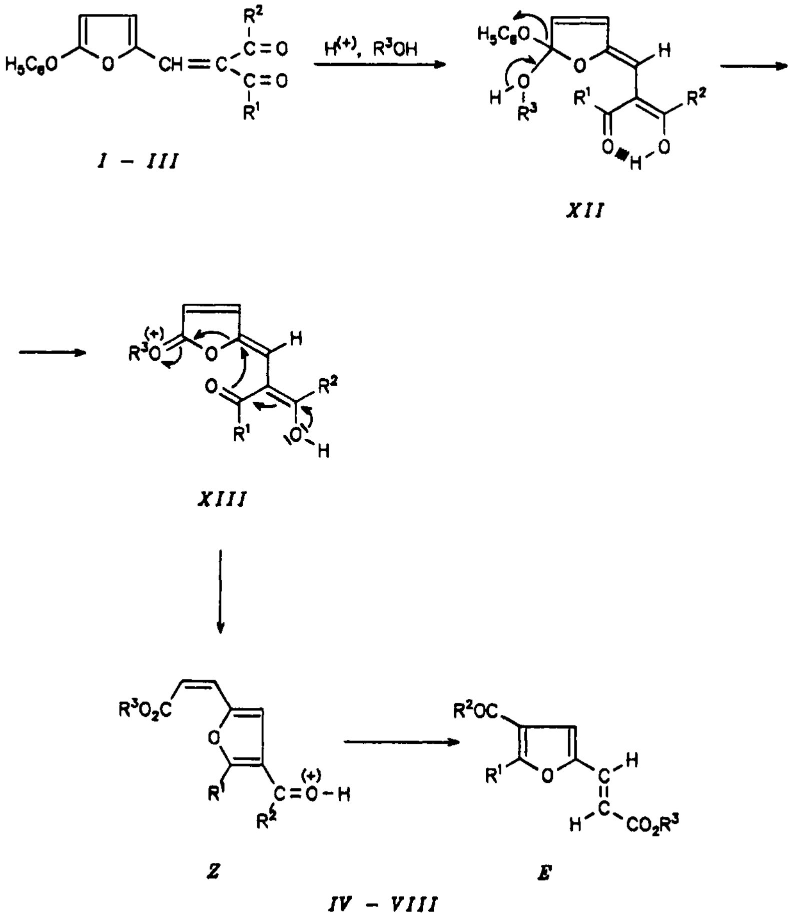
XI

Furan derivatives having the ester group in position 4 originated from the starting 2-(5-phenoxy-2-furylmethylene)-1,3-ketoesters *II* and *III*. No *Z* isomer could be identified in the crude product, the *E* isomer being the only compound isolated in 63% (*E-VI*), 48% (*E-VII*) and 45% (*E-VIII*) yields.

The structure of furans *IV* – *VIII* was verified both by elemental analyses (Table I) and spectral measurements – IR and UV data are listed in Table II, ^{1}H and ^{13}C NMR spectra in Tables III and IV, respectively. The mass spectral data are presented at the end of this contribution.

Base-catalyzed hydrolysis of the ester *E-IV* produced the known acid *E-IX* (ref.[10]). Hydrolysis of esters *Z-V* and *E-VI* afforded the corresponding acids *Z-IX* (yield 87%) and *E-X* (yield 66%).

This reaction constitutes a new rearrangement of the furan ring in acid medium. The simple one-step reaction made it possible to synthesize 2,4,5-trisubstituted furan derivatives from the easily accessible 2,5-disubstituted furans. The probable mechanism for formation of compounds *IV* – *VIII* is illustrated in Scheme 1. The first step involves protonization of oxygen at the acetyl group in the side chain. The following attack of the lone electron pair of the alcohol-oxygen in position 5 of the furan ring gave rise to the intermediate *XII*. The acid hydrolysis of 2-methoxyfuran was reported[11] to be accompanied by a preferential protonization in position 5 under formation of 2-buten-4-olide. The intermediate *XIII* originated then by elimination of phenol, the presence of which was evidenced by gas-liquid chromatography. The intermediate *XIII* is stabilized by opening the butenolide system and by forming the 2,4,5-trisubstituted furan. The reaction produced the *Z* isomer, which underwent isomerization to give the *E* isomer. Such an isomerization took place with the mixture of isomers *Z*-*V* and *E*-*V* and also with the pure isomer *Z*-*V* by ethanolic hydrogen chloride.



SCHEME 1

Compound *I* afforded 4-alkylidene-2-buten-4-olide on replacement of alcohol for water; this is in accordance with the mechanism suggested. Structure of the butenolide was evidenced by elemental analysis and spectral means.

The IR spectrum of compound *XI* showed two absorption bands associated with stretching vibrations of carbonyl groups at 1 784 and 1 759 cm^{-1}. The ^1H NMR spectrum displayed two characteristic features – a long-range coupling constant $^3J_{2,3}$ = 5.4 Hz and a signal at δ 16.94 – for 2-buten-4-olides[12,13] and 3-substituted 2,4-pentanediones in an enol form[14], respectively. The (*Z,Z*)-*trans* geometry of this molecule was evidenced by the NOE effect between the methyl groups at C-7 and the H-5 proton and also by comparison with the ^1H NMR spectrum of (*Z*)-4-acetonylidene-2-buten-4-olide[15]. The main stabilization factor of the enol form is supposed to be a strong intramolecular hydrogen bonding forming an effective stable 6-membered ring. Symmetry of this ring is seen in the ^1H NMR spectrum by the appearance of only one signal each for the CH_3 and C=O groups in the ^{13}C NMR spectrum at δ 24.4 and 191.8, respectively. Recently[16], the X-ray analysis disclosed that the molecule of compound *XI* in solid state is constituted of two planar fragments – 2-buten-4-olide and the enol form of 2,4-pentanedione – both being twisted by 61.4° | 2°| around the single C-5–C-6 bond.

EXPERIMENTAL

Melting points were measured on a Boetius micro hot-stage, the IR spectra (wave numbers in cm^{-1}) of chloroform solutions and the UV spectra (wavelengths in nm) of methanolic solutions were measured with UR 70 (Zeiss, Jena) and UV VIS (Zeiss, Jena) spectrophotometers, respectively. The ^1H and ^{13}C NMR

TABLE I
Substituted 3-(2-furyl)propenoates prepared from synthons *I* – *III*

Compound	Yield, %	M. p., °C (Solvent)	Formula (M. w.)	Calculated/Found	
				% C	% H
E-IV	70	106 – 107 (methanol)	$C_{11}H_{12}O_4$ (208.2)	63.44 63.25	5.82 5.78
Z-V	61	84 – 85 (ethanol)	$C_{12}H_{14}O_4$ (222.3)	64.84 64.74	6.36 6.53
E-VI	63	97 – 98 (methanol)	$C_{12}H_{14}O_5$ (238.3)	60.49 60.19	5.93 5.83
E-VII	48	67 – 68 (ethanol)	$C_{13}H_{16}O_5$ (252.3)	61.89 61.67	6.41 6.35
E-VIII	45	77 – 78 (methanol)	$C_{12}H_{13}ClO_5$ (272.7)	52.85 52.70	4.81 4.65

spectra of deuteriochloroform solutions containing tetramethylsilane as an internal reference (chemical shifts are given in ppm at the δ scale, coupling constants in Hz) were recorded with the respective Tesla BS 487 (80 MHz, CW mode) and Varian XL-300 (75.43 MHz, FT mode) instruments at 25 °C. The reaction course and purity of compounds were monitored by thin-layer chromatography on Silufol sheets, detection by UV_{254} light or by iodine vapours.

2-(5-Phenoxy-2-furylmethylene)-1,3-ketoesters *II* and *III*

These compounds were synthesized from 5-phenoxy-2-furaldehyde and ethyl 4-chloromethyl-3-oxobutanoate or 3-oxobutanoate according to ref.[7].

(*Z,E*)-*II*: yellow crystals, m.p. 83 – 85 °C (ethanol). For $C_{17}H_{16}O_5$ (300.2) calculated: 68.02% C, 5.33% H; found: 68.23% C, 5.59% H. IR spectrum (chloroform): 1 038 m, 1 246 s, 1 460 s, 1 510 s, 1 620 s, 1 649 s ($\nu_{(C=C)}$), 1 738 m ($\nu_{(C=O)}$), 3 047 w, 3 145 ($\nu_{(C=C-H)}$). UV spectrum: 227 (2.95), 348 (3.25). ^1H NMR spectrum: 1.13 – 1.50 m, 3 H (CH_3); 2.35 s, 3 H (CH_3); 4.02 – 4.50 m, 2 H (OCH_2); 5.50 – 5.75 m, 1 H (H-4); 6.75 – 7.75 m, 7 H (H-arom, H-3, H-β).

TABLE II
UV and IR spectra of esters *IV* – *VIII* and acids *IX*, *X*

Compound	UV spectrum[a]		IR spectrum ($CHCl_3$), cm^{-1}				
	λ_{max}, nm	log ε	ν(=C–H)	ν(C=O)	ν(C=C)	δ(C=CH)	
E-IV	223	3.20	3 080	1 708	1 641	1 540	972
	305	3.44	3 025	1 684	1 579	1 437	
Z-V	215	3.03	3 025	1 710	1 627	1 538	–
	310	3.28	3 018	1 679	1 578	1 428	
E-V	223	3.21	3 025	1 708	1 640	1 540	968
	305	3.45		1 680	1 580	1 410	
E-VI	214	3.19	3 038	1 710	1 644	1 545	970
	304	3.40	3 022		1 599	1 438	
E-VII	214	3.23	3 030	1 709	1 642	1 542	970
	304	3.43	3 020		1 600	1 430	
E-VIII	219	3.21	3 020	1 715	1 641	1 532	965
	300	3.35			1 600	1 432	
Z-IX[b]	218	3.08	–	1 680	1 618	1 538	970
	302	3.26			1 578	1 449	
E-IX[b]	221	3.13	–	1 675	1 630	1 542	978
	300	3.25			1 578	1 420	
E-X[b]	213	3.19	–	1 680	1 630	1 540	960
	303	3.39			1 592	1 440	

[a] Concentration 1 . 10^{-4} mol l^{-1}, methanol; [b] in KBr.

(*Z,E*)-*III*: orange oil. ^{1}H NMR spectrum: 1.28 t, 3 H (CH_3, *E*); 1.29 t, 3 H (CH_3, *Z*); 4.23 q, 2 H (OCH_2, *E*); 4.27 q, 2 H (OCH_2, *Z*); 4.40 s, 2 H (CH_2Cl, *E*); 4.43 s, 2 H (CH_2, *Z*); 5.55 d, 1 H (H-4, *E*, *J* = 3.6); 5.60 d, 1 H (H-4, *Z*, *J* = 3.6); 7.00 – 7.50 m, 14 H (H-arom, H-β, H-3).

3-(5-R^1-4-COR^2-2-furyl)propenoates *IV* – *VIII*

Hydrogen chloride in alcohol (10% solution, 3 ml) was added to a stirred and cooled (0 – 5 °C) solution of 5-phenoxy-2-furylmethylene derivative *I* – *III*. The originally yellow mixture turned colourless after about 5 min and crystals of the product become to separate. The mixture was kept cooled and stirred for 2 h, the precipitate was filtered off, washed with 5% sodium hydroxide and water and crystallized from a suitable solvent. A survey of esters thus obtained is seen in Table I, spectral data are listed in Tables II – IV. Ratio of the particular isomers was estimated from the ^{1}H NMR spectra of the crude mixture.

Ethyl (*E*)-3-(4-Acetyl-5-methyl-2-furyl)propenoate (*E-V*)

A solution of (*E,Z*)-*II* (1.0 g) in ethanolic hydrogen chloride (20 ml) was stirred in a closed flask at room temperature for 8 h, the solvent was distilled off under reduced pressure and the solid was crystallized from ethanol. Yield 0.8 g (80%) of colourless crystals, m.p. 83 – 84 °C. For $C_{12}H_{14}O_4$ (222.3) calculated: 64.84% C, 6.36% H; found: 64.96% C, 6.38% H.

TABLE III
^{1}H NMR spectra of esters *IV* – *VIII* and acids *IX*, *X* in chloroform at 25 °C

Compound	H-3	H-α	H-β[a]	R^1	R^2	R
E-IV	6.80 s	6.28 d	7.35 d	2.61 s	2.39 s	3.76 s
E-V	6.80 s	6.32 d	7.35 d	2.64 s	2.42 s	1.32 t 4.25 q
Z-V[b]	7.97 s	5.76 d	7.71 d	2.60 s	2.45 s	1.33 t 4.24 q
E-VI	6.85 s	6.30 d	7.36 d	2.63 s	4.31 q 1.35 t	3.78 s
E-VII	6.84 s	6.30 d	7.35 d	2.62 s	4.31 q 1.35 t	1.32 t 4.25 q
E-VIII	6.88 s	6.40 d	7.38 d	4.90 s	4.34 q 1.36 t	3.08 s
E-IX[c]	7.16 s	6.28 d	7.43 d	2.62 s	2.43 s	–
Z-IX[c]	7.99 s	5.85 d	6.84 d	2.58 s	2.40 s	–
E-X[c]	7.07 s	6.29 d	7.45 d	2.66 s	–	–

[a] *J*(α, β) 15.9; [b] *J*(a, β) 12.8; [c] in hexadeuterioacetone.

(*Z*)-3-(4-Acetyl-5-methyl-2-furyl)propenoic Acid (*Z-IX*)

A mixture of the ethyl ester *Z-V* (0.23 g) and KOH (0.11 g) in water (10 ml) was refluxed for 1 h, cooled and acidified with 18% hydrochloric acid to pH 1. The separated yellow precipitate was filtered off and crystallized from water. Yield 0.17 g (87%), m.p. 167 – 170 °C. For $C_{10}H_{10}O_4$ (194.2) calculated: 61.84% C, 5.20% H; found: 62.04% C, 5.22% H.

(*E*)-3-(4-Acetyl-5-methyl-2-furyl)propenoic Acid (*E-IX*)

Starting from methyl ester *E-IV*, the same procedure was applied to afford *E-IX* in 74% yield, m.p. 201 – 203 °C (ethanol–water; ref.[10] reported m.p. 205 °C). For $C_{10}H_{10}O_4$ (194.2) calculated: 61.84% C, 5.20% H; found: 61.52% C, 5.26% H.

(*E*)-3-(4-Carboxy-5-methyl-2-furyl)propenoic Acid (*E-X*)

The title product was prepared by hydrolysis of compound *E-VI* in 66% yield, m.p. 258 – 260 °C (ethanol–water). For $C_9H_8O_5$ (196.2) calculated: 55.10% C, 4.12% H; found: 55.01% C, 3.95% H.

TABLE IV
^{13}C NMR spectra (δ, ppm) and coupling constants (Hz) of esters *IV* – *VIII* and acids *IX*, *X* in deuteriochloroform

Compound	C=O	CO_2	C-5	C-4	C-3	C-2	C-α	C-β	R^1	R^2	R
E-IV	193.4	167.2	160.8	123.5	114.8	148.4	116.1	130.3	14.7	29.1	51.7 q
E-V	193.4	166.7	160.7	123.6	114.5	148.6	116.8	130.1	14.7	29.1	60.6 t 14.3 q
Z-V	194.2	165.9	159.7	123.6	115.2	148.5	117.5	130.0	14.5	29.0	60.3 t 14.3 q
E-VI	163.1	167.1	161.3	116.1	115.2	148.4	115.7	130.4	14.2	60.3 14.0	51.6 q
E-VII	163.1	166.6	161.2	115.0	115.0	148.5	116.1	130.1	14.2	60.3 14.0	60.4 t 14.2 q
E-VIII	162.1	166.9	156.4	118.5	114.7	150.6	118.3	129.5	35.3	61.1 14.2	51.9
E-IX	193.5	171.8	161.5	123.7	115.5	148.2	115.8	123.3	14.8	21.1	–
Z-IX	194.4	170.5	160.4	123.7	113.7	148.2	118.5	132.2	14.6	29.0	–
E-X	164.3	167.5	162.3	117.0	116.5	149.6	117.0	131.5	13.9	–	–

(4*Z*,6*Z*)-6-Acetyl-7-hydroxy-2,4,6-octatrien-4-olide (*XI*)

Dilute hydrochloric acid (18%, 2.5 ml) was added to a cooled (0 – 5 °C) and stirred solution of 3-(5-phenoxy-2-furylmethylene)-2,4-pentanedione (*I*) in ether (35 ml) during 30 min. The mixture was stirred for 4 h, the separated precipitate was filtered off, washed with cooled ether and water to neutral reaction and crystallized from benzene to furnish yellow crystals *XI*, 4.8 g, m.p. 101 – 105 °C. For $C_{10}H_{10}O_4$ (194.2) calculated: 61.84% C, 5.20% H; found: 61.86% C, 5.21% H. IR spectrum: 1 784 s, 1 759 s $(\nu_{(C=O)})$. UV spectrum: 298 (3.11), 316 (3.17), 435 (2.10). ^{1}H NMR spectrum: 2.16 s, 6 H (2 × CH_3); 5.91 s, 1 H (H-5); 6.22 dd, 1 H (H-2, $J_{2,3}$ = 5.4; $J_{5,2}$ = 0.8); 7.52 dd, 1 H (H-3, $J_{3,5}$ = 0.3); 16.94 s, 1 H (OH). ^{13}C NMR spectrum: 24.4 s (CH_3), 105.8 s (C-6 and C-7), 108.5 d (C-5), 119.2 d (C-2), 143.8 d (C-3), 148.8 s (C-4), 169.3 s (C-1), 191.8 s (CO).

Mass Spectra *m/z*, (Rel. Intensity, %)

E-IV: 208 ($M^{+\cdot}$, 100), 194 (9), 193 (65), 177 (44), 162 (7), 151 (32), 150 (6), 149 (6), 134 (30), 133 (6), 132 (9), 123 (7), 81 (9), 78 (5), 77 (10), 63 (11), 52 (7), 51 (11), 44 (12), 43 (94), 39 (6), 29 (7), 28 (47).

Z-V: 222 ($M^{+\cdot}$, 42), 207 (9), 194 (7), 193 (9), 177 (25), 152 (6), 150 (21), 137 (8), 135 (22), 77 (7), 63 (7), 52 (5), 51 (7), 43 (100), 29 (8), 28 (10), 27 (8).

E-VI: 239 ($M^{+\cdot}$, 13), 238 (97), 210 (14), 209 (72), 207 (41), 195 (5), 193 (29), 192 (10), 181 (10), 180 (8), 179 (26), 178 (11), 164 (5), 161 (6), 151 (15), 150 (8), 149 (13), 136 (10), 134 (5), 133 (11), 113 (6), 105 (6), 82 (9), 77 (10), 63 (9), 52 (6), 51 (8), 44 (27), 43 (36), 39 (6), 32 (9), 31 (6), 30 (6), 29 (15), 28 (100), 27 (9), 26 (6).

E-VII: 253 (15), 252 ($M^{+\cdot}$, 100), 224 (14), 223 (35), 208 (9), 207 (7), 206 (6), 196 (6), 195 (32), 180 (31), 179 (28), 178 (25), 152 (6), 151 (22), 149 (7), 137 (6), 136 (21), 134 (7), 133 (10), 105 (6), 81 (10), 78 (6), 77 (11), 63 (10), 52 (7), 51 (8), 44 (26), 32 (5), 31 (5), 31 (9), 29 (21), 28 (57), 27 (18), 26 (8).

E-V: 223 (8), 222 ($M^{+\cdot}$, 40), 208 (12), 195 (9), 194 (10), 180 (20), 179 (5), 178 (24), 152 (7), 151 (14), 138 (10), 135 (16), 134 (5), 133 (5), 81 (10), 79 (5), 78 (5), 77 (10), 61 (13), 57 (6), 55 (7), 53 (5), 52 (7), 51 (10), 50 (4), 43 (100), 41 (7), 39 (6), 29 (14), 28 (10), 27 (12).

E-VIII: 272 ($M^{+\cdot}$, 49), 244 (8), 242 (28), 241 (17), 240 (11), 239 911), 238 (83), 237 (97), 229 (5), 227 (13), 223 (6), 213 (6), 210 (17), 209 (100), 207 (33), 199 (5), 193 (31), 181 (21), 179 (22), 178 (11), 164 (10), 163 (5), 151 (15), 150 (11), 149 (28), 139 (5), 136 (5), 134 (6), 133 (10), 123 (5), 121 (8), 113 (19), 106 (6), 105 (10), 91 (6), 85 (8), 81 (8), 79 (7), 78 (6), 77 (21), 65 (7), 63 (17), 62 (5), 59 (13), 54 (7), 53 (10), 52 (8), 51 (25), 50 (8), 44 (22), 43 (28), 41 (5), 39 (8), 38 (5), 36 (17), 31 (8), 30 (6), 29 (19), 28 (53), 27 (13), 26 (8).

Z-IX: 195 (10), 194 ($M^{+\cdot}$, 100), 180 (9), 179 (80), 151 (10), 137 (29), 135 (5), 133 (9), 109 (10), 82 (7), 63 (5), 51 (6), 44 (7), 43 (79), 32 (7), 28 (26).

E-X: 198 (14), 196 ($M^{+\cdot}$, 100), 180 (17), 179 (11), 164 (17), 152 (20), 150 (26), 137 (8), 136 (38), 133 (10), 108 (8), 99 (11), 95 (8), 83 (8), 81 (8), 77 (15), 75 (8), 69 (13), 67 (10), 63 911), 57 (9), 55 (13), 53 (17), 52 (16), 51 (19), 45 (16), 43 (53), 41 (19), 39 (12), 28 (15), 27 (22).

XI: 195 ($M^{+\cdot}$, 6), 194 (55), 179 (16), 176 (5), 152 (21), 137 (12), 134 (28), 110 (6), 82 (6), 69 (6), 55 (12), 54 (8), 44 (8), 43 (100), 28 (48), 26 (6).

REFERENCES

1. Poirer J. M., Dujardin G.: Heterocycles *25*, 399 (1987).
2. Kametani T., Katoh T., Tsubuki M., Honda T.: J. Am. Chem. Soc. *108*, 7055 (1986).
3. Jefford C. W., Sledeski A. W., Boukouvalas J.: Tetrahedron Lett. *28*, 949 (1987).
4. Jefford C. W., Jaggi D., Boukovalas J.: Tetrahedron Lett. *28*, 4037 (1987).
5. Marini-Bettolo R., Flecker P., Tsai T. Y. R., Wiesner K.: Can. J. Chem. *59*, 1403 (1981).

6. Wulff W. D., Gilbertson S. R., Springer J. P.: J. Am. Chem. Soc. *108*, 520 (1986).
7. Marchalín Š., Ilavský D., Kováč J.: Z. Chem. *27*, 406 (1987).
8. Marchalín Š., Pavlíková F., Ilavský D.: Collect. Czech. Chem. Commun. *54*, 1336 (1989).
9. Marchalín Š., Ilavský D., Kováč J., Bruncko M.: Collect. Czech. Chem. Commun. *55*, 718 (1990).
10. Jiménez F., Roncero A. V.: An. Real. Soc. Espan. Fis. Quim., B *45*, 1591 (1949); Chem. Abstr. *46*, 7089a (1952).
11. Garst J. E., Schmir G. L.: J. Org. Chem. *39*, 2921 (1974).
12. Yamada K., Togawa Y., Kato T., Hirata Y.: Tetrahedron *27*, 5445 (1971).
13. Sorotskaya L. N., Badovskaya L. A., Kaklyugina T. Ya., Belenkii L. I., Ignatenko A. V., Krutošíková A., Panieva L. A.: Zh. Org. Khim. *25*, 175 (1989).
14. Yoshida Z., Ogoshi H., Tokumitsu T.: Tetrahedron *26*, 5691 (1970).
15. Nilsson M.: Acta Chem. Scand. *18*, 441 (1964).
16. Lokaj J., Sivý P., Ilavský D., Marchalín Š., Vrábel V., Kettmann V.: Acta Crystallogr., in press.

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