SOME NEW DERIVATIVES OF 2-PENTENEDIOIC ACID

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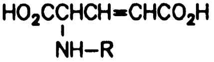
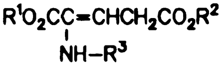
Michael addition of N-acylaminomalonates to propynoic esters led to the (*E*)-4-(N-acylamino)-4-carboxy-2-pentenedioic triesters *IIa* – *IIf*. On deprotection and decarboxylation, these compounds afforded 2-(N-acylamino)-2-pentenedioic acid derivatives *IIIa* – *IIIc*. Dimethyl (*E*)-4-diazo-2-pentenedioate (*IV*) was prepared by direct transfer of the diazo group. On treatment of *IV* with 4-toluenesulfonic acid, 2-(4-toluenesulfonyloxy)-2-pentenedioate (*V*) was formed. Iodination of (*E*)-2-pentenedioic acid led directly to the 2,4-diiodo derivative *VII*. From dimethyl (*E*)-4-oxo-2-pentenedioate the oximino ester *VIIIa* and the free oximino acid *VIIIb* were prepared.

In the course of our syntheses of diverse 3,4-unsaturated aliphatic amino acids[1], many attempts were carried out to prepare the yet unknown olefinic analogue of glutamic acid, i.e. 4-amino-2-pentenedioic acid (*Ia*). During these experiments, which all unfortunately failed in the final stage (most probably due to the apparent instability of *Ia* – *Ic*) some new derivatives of 2-pentenedioic acid were synthesized and characterized.



$$\mathrm{HO_2C\underset{NH-R}{\underset{|}{C}}HCH{=}CHCO_2H}$$



$$\mathrm{R^1O_2C\underset{NH-R^3}{\underset{|}{C}}{=}CHCH_2CO_2R^2}$$

Ia, $R = H$
Ib, $R = COCH_3$
Ic, $R = CHO$

IIIa, $R^1 = R^2 = H$; $R^3 = COCH_3$
IIIb, $R^1 = H$; $R^2 = C_2H_5$; $R^3 = COCH_3$
IIIc, $R^1 = R^2 = H$; $R^3 = CHO$
IIId, $R^1 = R^2 = CH_3$; $R^3 = COCH_3$

Addition of N-acylaminomalonic esters to propynoic acid esters, catalyzed by KF/18-crown-6 (ref.[2]), afforded the 4-(N-acylamino)-4-carboxy-2-pentenedioic acid triesters *IIa* – *IIf* in high yields (see Scheme 1). NMR analyses showed the compounds *IIa* – *IIf* to be exclusively of the *E* configuration (J = 15.6 – 15.9 Hz).

All attempts to convert the adducts *IIa* – *IIf* into the acids *Ib* and *Ic* either by basic hydrolysis (*IIa* – *IIc*) or by acidolysis (*IId* – *IIf*) gave, instead of them, the isomeric compounds *IIIa* – *IIIc* only. A mechanism for this kind of isomerisation had been suggested[3]. Prepared compounds *IIa* – *IIf* and *IIIa* – *IIIc* are listed in Table I.

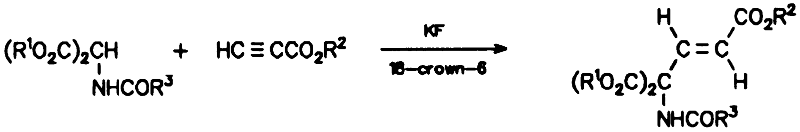
Further experiments were directed to the preparation of the 2-pentenedioic acid derivatives, suitably substituted at C-4. Thus, dimethyl (*E*)-4-diazo-2-pentenedioate (*IV*) was prepared from the unsubstituted diester by diazo group transfer[5], using 4-toluenesulfonylazide. Treatment of *IV* with 4-toluenesulfonic acid[6] in acetonitrile gave dimethyl 2-(4-toluenesulfonyloxy)-2-pentenedioate (*V*); as a byproduct, ester *IIId* was also isolated, which apparently arose from the reaction between *IV* and the solvent.

TABLE I
Triesters *IIa* – *IIf* and acids *IIIa* – *IIIc*

Compound	M. p., °C Yield, %	Formula (M. w.)	Calculated/Found		
			% C	% H	% N
IIa	110.5 – 111.5[a]	$C_{11}H_{15}NO_7$	48.35	5.53	5.13
	70	(273.2)	48.28	5.68	5.08
IIb	143 – 144.5[b]	$C_{14}H_{21}NO_7$	53.32	6.71	4.43
	90	(315.3)	53.53	6.80	4.32
IIc	oil[c]	$C_{14}H_{21}NO_7$	53.32	6.71	4.43
	86	(315.3)	53.20	6.81	4.34
IId	141 – 142[b]	$C_{18}H_{29}NO_7$	58.20	7.87	3.77
	89	(371.4)	58.49	8.00	3.69
IIe	127 – 129[d]	$C_{19}H_{31}NO_7$	59.20	8.11	3.63
	71	(385.5)	59.56	8.23	3.53
IIf	128 – 129[e]	$C_{20}H_{33}NO_7$	60.12	8.33	3.51
	81	(399.5)	60.09	8.40	3.42
IIIa	183 – 184[f, g]	$C_7H_9NO_5$	44.92	4.85	7.49
	46[h], 82[i]	(187.2)	44.85	5.01	7.35
IIIb	142 – 144[j]	$C_9H_{13}NO_5$	50.23	6.09	6.51
	91[k]	(215.2)	49.89	6.16	6.41
IIIc	147 – 149[l]	$C_6H_7NO_5$	41.62	4.08	8.09
	86[m]	(173.1)	41.42	4.09	8.18

[a] 2-Propanol–ether; [b] benzene–hexane; [c] ref.[4], b.p. 140 – 145 °C/5 Pa (but undistillable in our hands); [d] heptane; [e] hexane; [f] ethanol–petroleum ether 60 – 70 °C; [g] ref.[3], m.p. 175 – 176 °C; [h] from *IIa* or *IIc*, method *A*; [i] from *IIf*, method *B*; [j] ethanol–ether–petroleum ether; [k] from *IId*, method *B*; [l] water; [m] from *IIe*, method *B*.

Attempts were also made to prepare 4-halogeno derivatives of 2-pentenedioic acid. Reactions between *IV* and hydrogen chloride (or hydrogen bromide) gave only complicated and unseparable mixtures. The previously so-called "4-chloroglutaconic acid" (see ref.[7]) was identified by NMR analysis as the 2-chloro isomer *VI*. Iodination of (*E*)-2-pentenedioic acid by I_2/HIO_3 gave directly the 2,4-diiodo derivative *VII*, even when one equivalent of the iodination reagent was used.



IIa – IIf

IIa, $R^1 = R^2 = R^3 = CH_3$

IIb, $R^1 = R^3 = CH_3$; $R^2 = C(CH_3)_3$

IIc, $R^1 = R^2 = C_2H_5$; $R^3 = CH_3$

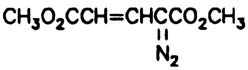
IId, $R^1 = C(CH_3)_3$; $R^2 = C_2H_5$; $R^3 = CH_3$

IIe, $R^1 = R^2 = C(CH_3)_3$; $R^3 = H$

IIf, $R^1 = R^2 = C(CH_3)_3$; $R^3 = CH_3$

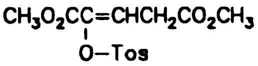
SCHEME 1

Finally, dimethyl (*E*)-4-oxo-2-pentenedioate[8] was converted into the oximino ester *VIIIa*, which was then saponified to the free acid *VIIIb*. None of these two compounds could be successfully reduced to *Ia*.



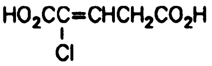
$\mathrm{CH_3O_2CCH{=}CH\underset{\underset{N_2}{\|}}{C}CO_2CH_3}$

IV



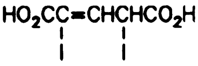
$\mathrm{CH_3O_2C\underset{\underset{O-Tos}{|}}{C}{=}CHCH_2CO_2CH_3}$

V



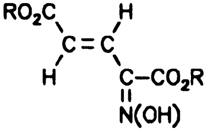
$\mathrm{HO_2C\underset{\underset{Cl}{|}}{C}{=}CHCH_2CO_2H}$

VI



$\mathrm{HO_2C\underset{\underset{I}{|}}{C}{=}CH\underset{\underset{I}{|}}{C}HCO_2H}$

VII



VIIIa, R = CH_3

VIIIb, R = H

EXPERIMENTAL

Melting points were determined on a Büchi SMP-20 apparatus and were uncorrected. NMR spectra were measured on the Jeol FX-60 FT NMR spectrometer (59.797 and 15.036 MHz for ^{1}H and ^{13}C, respectively) at 25 °C in the solvents indicated; tetramethylsilane served as an internal standard. Carbon signal multiplicities were determined from SFORD (Single Frequency Off-Resonance Decoupling) spectra. Chemical shifts are given in ppm (δ-scale), coupling constants (*J*) in Hz.

(*E*)-4-(N-Acylamino)-4-carboxy-2-pentenedioic Esters (*IIa* – *IIf*)

18-Crown-6 (130 mg, 0.5 mmol) and dry potassium fluoride (120 mg, 2.1 mmol) were added to a solution (or suspension) of the N-acylaminomalonic acid diester (10 mmol) in dry acetonitrile (10 ml). After 10 min stirring, the propynoic acid ester (11 mmol) was slowly dropped in under cooling with tap water. The mixture was then stirred at room temperature overnight (for *IIe* and *IIf*, the mixture was refluxed for 2 h), evaporated to dryness and the residue was dissolved in ethyl acetate (30 ml). The coloured solution was then repeatedly washed with saturated solution of sodium bicarbonate and water (until the aqueous washings were almost colourless), dried with sodium sulfate and evaporated. Alternatively (as used for *IIb*), the reaction mixture was poured into cold water (50 ml), the product was filtered, washed well with cold water and dried. The crude product was recrystallized, if necessary, from the solvent indicated in Table I.

Compound IIa, ^{1}H NMR spectrum ($CDCl_3$): 2.10 s, 3 H (CH_3CO); 3.75 s, 3 H ($COOCH_3$); 3.83 s, 6 H (2 × $COOCH_3$); 5.94 and 7.47, AB (2 × =CH–, *J*(A,B) = 15.9); 7.00 brs, 1 H (N–H). ^{13}C NMR spectrum ($CDCl_3$): 22.7 q, 51.9 q, 54.2 q (2 C), 66.7 s, 122.5 d, 140.9 d, 165.8 s, 166.4 s, 169.0 s (2 C).

Compound IIb, ^{1}H NMR spectrum ($CDCl_3$): 1.47 s, 9 H ($(CH_3)_3C$); 2.10 s, 3 H (CH_3CO); 3.83 s, 6 H (2 × $COOCH_3$); 5.84 and 7.32, AB (2 × =CH–, *J*(A,B) = 15.9); 6.97 s, 1 H (N–H). ^{13}C NMR spectrum ($CDCl_3$): 22.7 q, 28.1 q (3 C), 54.1 q (2 C), 66.7 s, 124.6 d, 139.3 d, 164.6 s, 166.6 s, 168.9 s, 169.1 s.

Compound IIc, ^{1}H NMR spectrum ($CDCl_3$): 1.29 t, 9 H (3 × $COOCH_2CH_3$, *J* = 7.3); 2.10 s, 3 H (CH_3CO); 4.21 q, 2 H ($COOCH_2CH_3$, *J* = 7.3); 4.29 q, 4 H (2 × $COOCH_2CH_3$, *J* = 7.3); 5.94 and 7.47, AB (2 × =CH–, *J*(A,B) = 15.9); 7.02 brs, 1 H (N–H). ^{13}C NMR spectrum ($CDCl_3$): 14.0 q (2 C), 14.3 q, 22.7 q, 61.0 t (2 C), 63.5 t, 66.9 s, 122.8 d, 140.9 d, 165.6 s, 166.1 s (2 C), 168.9 s.

Compound IId, ^{1}H NMR spectrum ($CDCl_3$): 1.29 t, 3 H ($COOCH_2CH_3$, *J* = 7.3); 1.48 s, 18 H (2 × $(CH_3)_3C$); 2.08 s, 3 H (CH_3CO); 4.19 q, 2 H ($COOCH_2CH_3$, *J* = 7.3); 5.90 and 7.40, AB (2 × =CH–, *J*(A,B) = 15.9); 6.89 brs, 1 H (N–H). ^{13}C NMR spectrum ($CDCl_3$): 14.2 q, 22.8 q, 27.7 q (6 C), 60.7 t, 67.5 s, 84.3 s (2 C), 122.2 d, 141.6 d, 164.9 s (2 C), 165.7 s, 168.5 s.

Compound IIe, ^{1}H NMR spectrum ($CDCl_3$): 1.47 s, 27 H (3 × $(CH_3)_3C$); 5.84 and 7.27, AB (2 × =CH–, *J*(A,B) = 15.9); 7.04 s, 1 H (N–H); 8.23 s, 1 H (COOH). ^{13}C NMR spectrum ($CDCl_3$): 27.7 q (3 C), 28.1 q (6 C), 67.1 s, 84.6 s, 124.4 d, 139.3 d, 159.3 d, 164.6 s, 177.5 s (2 C).

Compound IIf, ^{1}H NMR spectrum ($CDCl_3$): 1.49 s, 27 H (3 × $(CH_3)_3C$); 2.10 s, 3 H (CH_3CO); 5.82 and 7.27 d, AB (2 × =CH–, *J*(A,B) = 15.6); 6.87 brs, 1 H (N–H). ^{13}C NMR spectrum ($CDCl_3$): 22.9 q, 27.7 q (6 C), 28.1 q (3 C), 65.1 s, 80.8 s, 84.2 s (2 C), 123.8 d, 140.2 d, 165.0 s, 177.4 s, 178.2 s (2 C).

2-(N-Acylamino)-2-pentenedioic Acid Derivatives (*IIIa* – *IIIc*)

A) Saponification of *IIa* or *IIc*. The title compound (10 mmol) was dissolved in methanol (20 ml), 2.5 M aqueous sodium hydroxide (16 ml; 40 mmol) was added and the solution was stirred overnight. Methanol was evaporated in vacuo, water (15 ml) was added to dissolve the suspended salt and the solution was stirred with moist Dowex 50 × 8 (100 – 200 mesh; H$^+$-cycle; 40 ml) for 3 h. The filtrate was evaporated and the crude *IIIa* recrystallized.

B) Acidolysis of *IId* – *IIf*. The compound (2 mmol) was dissolved in trifluoroacetic acid (5 ml), allowed to stand at laboratory temperature for 2 h, evaporated in vacuo, dry ether (5 ml) added and evaporated again. The crude *IIIa* – *IIIc* were recrystallized, as shown in Table *I*.

Compound IIIa, ^{1}H NMR spectrum (CD_3SOCD_3): 1.97 s, 3 H (CH_3CO); 3.13 mt, 2 H; 6.80 mt, 1 H. ^{13}C NMR spectrum (CD_3SOCD_3): 22.6 q, 34.0 t, 128.9 s, 133.8 d, 171.1 s, 174.9 s, 175.1 s.

Compound IIIb, ^{1}H NMR spectrum (CD_3COCD_3): 1.25 t, 3 H (CH_3CH_2O, J = 7.3); 2.08 s, 3 H (CH_3CO); 3.25 d, 2 H (J = 7.2); 4.14 q, 2 H (CH_3CH_2O, J = 7.3); 6.32 t, 1 H (=CH–, J = 7.2); 8.45 brs, 1 H (N–H). ^{13}C NMR spectrum (CD_3COCD_3): 14.4 q, 22.9 q, 34.4 t, 61.2 t, 128.8 d, 128.9 s, 165.7 s, 168.9 s, 170.9 s.

Compound IIIc, ^{1}H NMR spectrum ($CDCl_3$ + CD_3OD, 4 : 1): 3.20 d, 2 H (J = 7.0); 6.96 t, 1 H (=CH–, J = 7.0); 8.15 s, 1 H. ^{13}C NMR spectrum ($CDCl_3$ + CD_3OD, 4 : 1): 34.0 t, 125.8 s, 129.4 d, 159.8 d, 165.2 s, 172.0 s.

Dimethyl (*E*)-4-Diazo-2-pentenedioate (*IV*)

Triethylamine (15.2 g, 0.15 mol) in dry ether (45 ml) was added dropwise during 2 h to a stirred solution of dimethyl (*E*)-2-pentenedioate (23.7 g, 0.15 mol) and 4-toluenesulfonylazide (29.6 g, 0.15 mol) in dry ether (150 ml), cooled to 5 °C. Stirring and cooling was then continued for 1.5 h (the bath temperature rose to 10 °C during this period), the orange mixture was poured into hexane (500 ml) and kept overnight at 5 °C. The filtered solution was evaporated, the residue dissolved in benzene (50 ml) and chromatographed on a silica gel column (42 x 2.5 cm, eluted with benzene). Yield, 18.5 g (67%) of compound *IV*, m.p. 69 – 72 °C; from petroleum ether (30 – 40 °C), m.p. 72 – 73 °C. ^{1}H NMR spectrum ($CDCl_3$): 3.76 s, 3 H ($COOCH_3$); 3.86 s, 3 H ($COOCH_3$); 5.76 d, 1 H (=CH–, J = 15.9); 7.35 d, 1 H (=CH–, J = 15.9). ^{13}C NMR spectrum ($CDCl_3$): 51.6 q, 52.6 q, 111.4 d, 127.6 s, 131.0 d, 163.6 s, 166.5 s. For $C_7H_8N_2O_4$ (184.2) calculated: 45.64% C, 4.38% H, 15.22% N; found: 45.66% C, 4.20% H, 15.15% N.

Dimethyl 2-(4-Toluenesulfonyloxy)-2-pentenedioate (*V*)

Ester *IV* (1.84 g, 10 mmol) was added during 5 min to a solution of 4-toluenesulfonic acid (1.89 g, 11 mmol) in dry acetonitrile (10 ml). The mixture, from which nitrogen was evolved was stirred at laboratory temperature for 1 h, evaporated in vacuo, taken into ethyl acetate (30 ml), washed with a little sodium hydrogen carbonate solution, water and dried. After evaporation, the dark yellow oil was dissolved in benzene (1 ml) and flash-chromatographed on a silica gel column (26 x 1.5 cm). On elution with benzene, containing 1% of methanol, two products were isolated.

Compound V: oily, slowly solidified, 700 mg (21%), m.p. 50 – 54 °C; from benzene–hexane, m.p. 56 – 58 °C. ^{1}H NMR spectrum ($CDCl_3$): 2.44 s, 3 H (=C–CH_3); 3.28 d, 2 H (CH_2, J = 7.3); 3.64 s, 3 H ($COOCH_3$); 3.70 s, 3 H ($COOCH_3$); 6.91 t, 1 H (=CH–, J = 7.3); 7.36 and 7.85, AA'BB', 4 H (Tos, ΣJ = 8.5). ^{13}C NMR spectrum ($CDCl_3$): 21.1 q, 31.2 t, 51.6 q, 51.9 q, 127.2 d, 127.9 d (2 C), 129.3 d (2 C), 132.3 s, 137.8 s, 148.3 s, 160.9 s, 168.9 s. For $C_{14}H_{16}O_7S$ (328.3) calculated: 51.21% C, 4.91% H, 9.76% S; found: 51.38% C, 4.77% H, 9.51% S.

Compound IIId: crystalline, 340 mg (16%), m.p. 97 – 101 °C; from benzene–petroleum ether (40 – 60 °C), m.p. 103 – 104 °C. ^{1}H NMR spectrum ($CDCl_3$): 2.13 s, 3 H (CH_3CO); 3.25 d, 2 H (CH_2, J = 7.3); 3.72 s, 3 H ($COOCH_3$); 3.80 s, 3 H ($COOCH_3$); 6.88 t, 1 H (=CH–, J = 7.3); 7.31 brs, 1 H (N–H). ^{13}C NMR spectrum ($CDCl_3$): 23.5 q, 34.3 t, 52.1 q, 52.6 q, 126.8 s, 127.7 d, 164.8 s, 170.9 s. For $C_9H_{13}NO_5$ (215.2) calculated: 50.23% C, 6.09% H, 6.51% N; found: 50.17% C, 6.20% H, 6.31% N.

Dimethyl (*E*)-4-Oximino-2-pentenedioate (*VIIIa*)

The starting compound, dimethyl 4-oxo-2-pentenedioate[8], m.p. 60 – 61 °C, was characterized spectroscopically and found to posses *E* configuration. ^{1}H NMR spectrum ($CDCl_3$): 3.84 s, 3 H ($COOCH_3$); 3.94 s, 3 H ($COOCH_3$); 6.98 d, 1 H (=CH–, *J* = 16.0); 7.63 d, 1 H (=CH–, *J* = 16.0). ^{13}C NMR spectrum ($CDCl_3$): 52.5 q, 53.2 q, 134.2 d, 135.4 d, 161.1 s, 165.1 s, 182.2 s. Hydroxylamine hydrochloride (4.17 g, 60 mmol) was dissolved in methanol (40 ml), dimethyl (*E*)-4-oxo-2-pentenedioate (5.16 g, 30 mmol) was added and the solution refluxed for 4 h. After evaporation in vacuo, the residue was extracted with ethyl acetate (3 × 20 ml), the extracts were washed with a little water, dried with magnesium sulfate and evaporated. The crude product was taken into benzene (16 ml), filtered and hot petroleum ether (fraction 60 – 70 °C, 20 ml) was added. The separated crystals were washed with petroleum ether; yield, 4.55 g (81%), m.p. 108 – 110 °C. The sample for analysis had m.p. 112 – 115 °C (benzene–petroleum ether). ^{1}H NMR spectrum ($CDCl_3$): 3.82 s, 3 H ($COOCH_3$); 3.91 s, 3 H ($COOCH_3$); 7.08 and 7.70, AB (2 × =CH–, *J*(A,B) = 16.5). ^{13}C NMR spectrum ($CDCl_3$): 52.1 q, 52.9 q, 126.7 d, 129.2 d, 162.6 s, 166.8 s, 176.9 s. For $C_7H_9NO_5$ (187.2) calculated: 44.92% C, 4.85% H, 7.49% N; found: 44.94% C, 4.94% H, 7.43% N.

(*E*)-4-Oximino-2-pentenedioic Acid (*VIIIb*)

The ester *VIIIa* (1.12 g, 6 mmol) and 1 M sodium hydroxide (18 ml) was stirred at 70 °C for 10 min, cooled, acidified with 5 M hydrochloric acid (3.5 ml), extracted with ethyl acetate (3 × 15 ml), dried and evaporated to give 0.90 g (95%) of *VIIIb*, m.p. 160 – 161 °C; from ethyl acetate–petroleum ether, m.p. 162 – 163 °C. ^{1}H NMR spectrum (CD_3OD): 6.96 and 7.67, AB (2 × =CH–, *J*(A,B) = 16.5). ^{13}C NMR spectrum (CD_3OD): 128.5 d, 129.3 d, 146.5 s, 165.8 s, 170.0 s. For $C_5H_5NO_5$ (159.1) calculated: 37.74% C, 3.17% H, 8.81% N; found: 37.74% C, 3.40% H, 8.54% N.

2-Chloro-2-pentenedioic Acid (*VI*)

The compound, prepared according to ref.[7] in 32% overall yield, was identified and characterized by its NMR spectra, which fully confirmed the structure *VI*; m.p. 152 – 155 °C (ref.[9], m.p. 156 – 157 °C). ^{1}H NMR spectrum (CD_3OD): 3.41 d, 2 H (CH_2, *J* = 6.8); 7.27 t, 1 H (=CH–, *J* = 6.8). ^{13}C NMR spectrum (CD_3OD): 35.6 t, 135.4 d, 142.7 s, 165.2 s, 173.1 s.

2,4-Diiodo-2-pentenedioic Acid (*VII*)

(*E*)-2-Pentenedioic acid (16.25 g; 125 mmol) and iodine (12.7 g; 50 mmol) were dissolved in ethanol (100 ml), iodic acid (11.0 g; 62.5 mmol) in water (25 ml) was added and the mixture was vigorously stirred for 20 h. The resulting dark yellow solution was evaporated in vacuo, the residue was taken into warm chloroform (20 ml) and the turbid solution was decanted from the dark-coloured impurities, which were extracted with a second portion of chloroform (10 ml). The combined chloroform solutions were cooled for 3 days at –15 °C, the product was filtered, washed well with cold chloroform and dried to give 14.43 g (30%) of yellow *VII*, m.p. 140 – 141 °C. The mother liquor was evaporated and the residue taken into dichloromethane (10 ml) to give, after freezing, another portion of *VII*, amounting 2.52 g (5%), m.p. 142 – 144 °C. Total yield, 16.95 g (35%); due to its instability in solution, the compound could not be purified for analysis. ^{1}H NMR spectrum (CD_3OD): 5.23 d, 1 H; (=CH–, *J* = 9.8); 7.55 d, 1 H (=CH–, *J* = 9.8). ^{13}C NMR spectrum (CD_3OD): 22.5 d, 99.5 s, 148.5 d, 165.7 s, 172.1 s. For $C_5H_4I_2O_4$ (381.9) calculated: 15.72% C, 1.06% H, 66.46% I; found: 16.55% C, 1.24% H, 64.40% I.

REFERENCES

1. Tolman V., Sedmera P.: Tetrahedron Lett. *29*, 6183 (1988).
2. Belsky I.: J. Chem. Soc., Chem. Commun. *1977*, 237.
3. Kishida Y., Terada A.: Chem. Pharm. Bull. *17*, 2417 (1969).
4. Birkofer L., Hempel K.: Chem. Ber. *96*, 1373 (1963).
5. Regitz M.: Angew. Chem., Int. Ed. Engl. *6*, 733 (1967).
6. Reichstein T., Schindler W.: Helv. Chim. Acta *23*, 669 (1940).
7. Urushibara Y.: Bull. Chem. Soc. Jpn. *3*, 200 (1928).
8. Corey E. J., Tramontano A.: J. Am. Chem. Soc. *103*, 5599 (1981).
9. Le Corre M.: Ann. Chim. (Paris) *3*, 193 (1968).

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