PREPARATION OF 2,2-DIMETHYL-5-R-1,3-DIOXANE-4,6-DIONE DERIVATIVES

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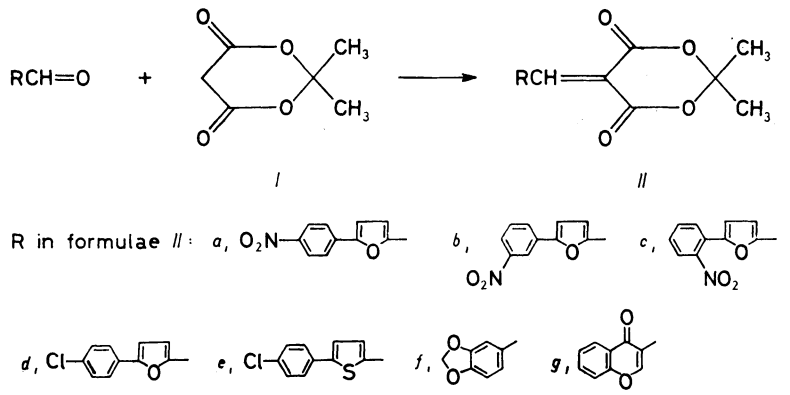
From the synthetic viewpoint, Meldrum's acid[1] (*I*) and its derivatives serve most often in preparation of arylidene condensation products[2], i.e. compounds that have a strongly polarized double bond. We have now synthesized new derivatives *II* (Scheme 1) by reaction of Meldrum's acid (*I*) with the corresponding aldehydes



SCHEME 1

EXPERIMENTAL

Melting points were measured on a Kofler block and are uncorrected. IR spectra (cm^{-1}) were measured by the KBr technique (Table II). Meldrum's acid (*I*) was prepared according to ref.[1] in 63% yield; m.p. 94—95°C (acetone) (reported[1] m.p. 94—95°C), reflux in chloroform solution being the method of choice. In most cases the products separated already during the reaction.

2,2-Dimethyl-5-R-1,3-dioxane-4,6-diones (*IIa—IIg*)

Acetic acid (0·2 ml) and piperidine (0·1 ml) were added to a stirred solution of *I* (1·44 g, 0·01 mol) and the corresponding aldehyde (0·011 mol) in chloroform (10 ml). After reflux for 1—2 h, the reaction mixture was cooled and set aside overnight. The separated precipitate of compound *II*

TABLE I

2,2-Dimethyl-5-R-1,3-dioxane-4,6-diones (*IIa*—*IIg*)

Compound Yield, %	Formula M.w.	M.p., °C Solvent[a]	Calculated/Found		
			% C	% H	% N
IIa	$C_{17}H_{13}ON_7$	224—225	59·47	3·82	4·08
74	343·3	E	59·52	3·91	4·20
IIb	$C_{17}H_{13}NO_7$	196—198	59·47	3·82	4·08
74	343·3	E	59·25	3·76	3·98
IIc	$C_{17}H_{13}NO_7$	160—161	59·47	3·82	4·08
59	343·3	E	59·58	3·96	4·15
IId	$C_{17}H_{13}ClO_5$	107—108	61·36	3·94	—
32	332·7	[b]	61·42	4·01	—
IIe	$C_{17}H_{13}ClSO_4$	187—188	58·53	3·75	—
58	348·8	E	58·69	3·82	—
IIf	$C_{14}H_{12}O_6$	171—172	60·86	4·38	—
83	272·2	CH–E	60·80	4·36	—
IIg	$C_{16}H_{11}O_6$	193—194	67·60	4·26	—
63	284·3	CH	67·51	4·23	—

[a] E ethanol, CH chloroform, CH–E (1 : 1); [b] purified by column chromatography (silica gel, benzene–acetone 9 : 1) and crystallization from ethanol.

TABLE II

IR spectra ($\tilde{\nu}$, cm^{-1}, KBr pellet) of derivatives *IIa*—*IIg*

Compound	C=O	C=C	NO_2	C—O	Other bands
IIa	1 734 1 728	1 635 1 628	1 528 1 385	1 035	1 718, 1 701, 1 653, 1 645, 1 076
IIb	1 795 1 713	1 580 1 572	1 528 1 345	1 043 1 034	1 560, 1 394, 1 269, 1 200
IIc	1 711	1 572	1 535 1 369	1 043 1 032	1 383, 1 284, 1 255, 1 232, 1 215, 1 204
IId	1 720	1 589	—	1 034 1 011	1 300, 1 269, 1 204, 1 092
IIe	1 734 1 705	1 564	—	1 032 1 005	1 391, 1 385, 1 300, 1 198, 1 080
IIf	1 711	1 562 1 555	—	1 099 1 034	1 455, 1 186
IIg	1 722	1 663 1 603	—	1 034 1 009	1 288, 1 232

was collected, washed with cold chloroform, dried and purified by crystallization or column chromatography (Table I).

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REFERENCES

1. Davidson D., Bernhard S. A.: J. Am. Chem. Soc. *70*, 3426 (1948).
2. Schuster P., Polansky O. E., Wessely F.: Monatsh. Chem. *95*, 53 (1964).

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