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NEW 2,4- AND 4,5-DIALKYLOXAZOLES

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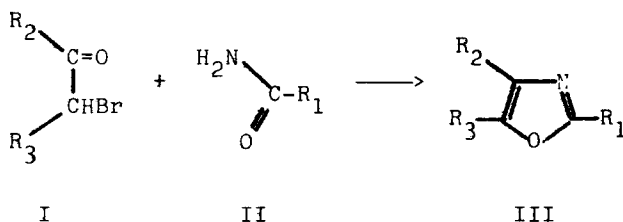
(By James A. Moore, Associate Editor)

NEW 2,4- AND 4,5-DIALKYLOXAZOLES

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(1/19/73) and V. G. Kartev

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New 2,4- and 4,5-dialkyloxazoles (III), free from isomeric contaminants (by spectral and glc data) have been prepared by the reaction¹⁻⁴ of amides (I) with α -bromoketones (II) obtained by the bromination of the corresponding ketones with dioxane dibromide.⁵



EXPERIMENTAL

Bromination of Ketones. - Dioxane dibromide (1 mole) was added slowly with stirring to an equimolar quantity of the ketone in 250 ml. of ether. The reaction mixture was then

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washed twice with water, several times with saturated aqueous potassium carbonate and twice again with water. The colorless solution thus obtained was dried (MgSO_4), the solvent evaporated and the residue distilled in vacuo. The yields and data of the α -bromoketones are given in Table I.

TABLE I. α -BROMOKETONES (II)^a

R_2	R_3	Yield	bp °C/mm
CH_3	$n\text{-C}_3\text{H}_7$	46%	45-99/10
CH_3	$i\text{-C}_3\text{H}_7$	57%	56-59/12
CH_3	$n\text{-C}_4\text{H}_9$	74%	65-68/14
CH_3	$n\text{-C}_6\text{H}_{13}$	75%	68-72/10
Me_3C	H	65%	68-73/8

^aAll new compounds gave satisfactory elemental analyses (within ± 0.30).

Oxazole Formation. A mixture of 0.2 mole of bromoketone (II) and of 0.5 mole of amide was heated in an oil bath (110-120°). After cooling, the mixture was made alkaline with 40% potassium hydroxide and extracted several times with ether. The extract was dried (MgSO_4), the ether was evaporated, the residue was heated for 20 min. with 1 g. of potassium hydroxide in a boiling water bath and the oxazole was distilled in vacuo. The yields and other data are given in Table II. All the oxazoles showed only end absorption in the UV (λ_{max} 212-222 $\text{m}\mu$, $\log \epsilon$ 3.60-3.84).

TABLE II. PREPARATION OF OXAZOLES (III)^{a,b}

R ₁	R ₂	R ₃	I (moles)	II	Yield (%)	Bp (°) (mm)	n _D ²⁰
H	Me ₃ C	H	0.60	0.15	27 ^b	128-130	1.4440
H	CH ₃	n-C ₃ H ₇	6.10	0.46	21	150-152	1.4444
H	CH ₃	i-C ₃ H ₇	4.10	0.30	22	143-145	1.4438
H	CH ₃	n-C ₄ H ₉	1.00	0.01	21	49-51/7	1.4468
H	CH ₃	n-C ₆ H ₁₃	4.50	0.27	22	73-75/8	1.4480
CH ₃	Me ₃ C	H	0.60	0.12	43 ^b	146-148	1.4430
n-C ₃ H ₇	CH ₃	H	0.75	0.30	20	57/20	1.4421
n-C ₅ H ₁₁	CH ₃	H	0.50	0.20	23	88/22	1.4452

^aThe reaction time was 3 hrs. except as otherwise noted.

^bTime: 5-6 hrs.

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