point is in agreement with the reported value), and 1-acetoxycyclohexanecarboxylic acid anilide with mp 130-132° (purified by chromatography on Al_2O_3 in $CHCl_3$). IR spectrum of the latter: 3400, 3280, 1720, 1667, 1609, 1540, and 1510 cm^{-1}.

The compositions of the synthesized compounds were confirmed by the results of elementary analysis.

SYNTHESIS OF 3,4,4-TRINITRO-Δ^1-PYRAZOLINES

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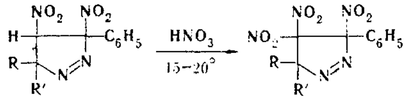
UDC 547.772.2'773'778.2.07

We have found that 3,4-dinitro-Δ^1-pyrazolines with a labile hydrogen atom in the 4 position of the pyrazoline ring are converted to 3,4,4-trinitro-Δ^1-pyrazolines (I-IV) when they are treated with nitric acid (sp. gr. 1.51) at 15-20° for 5-10 h. The products slowly decompose at 20-25° with the evolution of nitrogen oxides to give resinous products. The PMR spectra of I-IV do not contain the signal of a CH group.



I-IV

I $R=R'=C_6H_5$; II R and R' = diphenylene; III $R=C_6H_5$, $R'=p\text{-}BrC_6H_4$;

IV $R=CH_3$, $R'=p\text{-}NO_2C_6H_4$

The following compounds were obtained: I, mp 72°, 97% yield; II, mp 116°, 91% yield; III, mp 79°, 93% yield; IV, mp 63°, 94% yield. The compounds melt with decomposition. They were purified by reprecipitation from acetone solution by the addition of water.

The results of elementary analysis of I-IV were in agreement with the calculated values.

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SYNTHESIS OF SUBSTITUTED 6H-DIBENZO[b,d]PYRAN-6-ONE

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and L. V. Efimova

UDC 547.814.07:543.422.4'544

We have shown that intramolecular substitution of the o-nitro group to give 6H-dibenzo[b,d]pyran-6-one derivatives (IIa-d) occurs when o-nitrodiphenylcarboxylic acids (Ia-d) are refluxed in quinoline.

A solution of 0.005 mole of the appropriate acid I was refluxed in 20 ml of quinoline, after which the mixture was cooled and treated with 10% sodium carbonate solution. The sodium carbonate extract was acidified to pH 5 with hydrochloric acid, and the precipitate was removed by filtration, washed with water, and dried to give 6H-dibenzo[b,d]pyran-6-ones II. The following compounds were obtained: (reaction time, yield, and melting point given): IIa,

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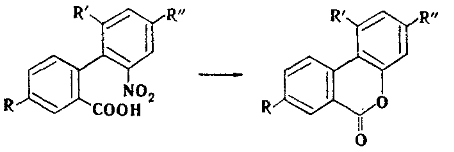
Scientific-Research Institute of Organic Intermediates and Dyes, Moscow 109388. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 5, pp. 703-704, May, 1977. Original article submitted December 3, 1976.



I a-d   II a-d

I,IIa,d R=R''=NO$_2$; b R=NO$_2$; c R=R''=H; a,c R'=COOH; b,d R'=H

30 min, 97%, 238-238.5° (dec., from 5% hydrochloric acid); IIb, 6 h, 42%, 246-247° (dec., from benzene); IIc, 6 h, 34%, 218.5-219° (dec., from benzene); IId, 1 h, 85%, 210-211° (from acetone). Compound IId was isolated from the reaction mixture by pouring into hydrochloric acid. The results of elementary analysis of IIa-d were in agreement with the calculated values. The individuality of the compounds was evaluated by thin-layer chromatography on Silufol (the spots had blue fluorescence in UV light). The band of the stretching vibrations of the lactone C=O group in the IR spectra of II lies at 1750-1760 cm^{-1} and the carboxyl group C=O band is observed at 1695-1715 cm^{-1}.

SYNTHESIS OF 4H-1,3-OXAZIN-4-ONIUM SALTS

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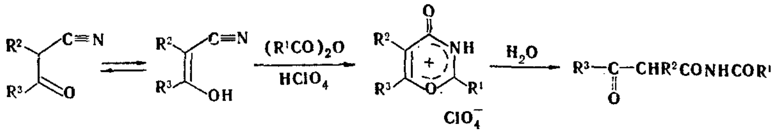
UDC 547.867.2.07

We have obtained the previously unknown 4H-1,3-oxazin-4-onium perchlorates (I) by acylation of β-ketonitriles with aliphatic acid anhydrides in the presence of equimolar amounts of 70% perchloric acid:



I   II

The IR spectra of I contain the following bands: an intense band at 1750 cm^{-1} (C=O) and a band of medium intensity at 1520 cm^{-1} $O{=}\overset{+}{C}{=}N$ fragment), which are characteristic for 4H-1,3-benzoxazin-4-onium salts, and a strong band at 1650 cm^{-1} (probably the stretching vibrations of a carbon-carbon double bond).

The action of water on perchlorates I gives β-keto acid N-acylamides: IIa, mp 103-104° (from benzene; in agreement with the literature value); IIb, mp 119-120° (from benzene); IIc,

TABLE 1

Compound	R¹	R²	R³	mp, °C	Yield, %
Ia	CH_3	H	C_6H_5	165	70
Ib	C_2H_5	H	C_6H_5	142—144	50
Ic	C_3H_7	H	C_6H_5	157—159	55
Id*	CH_3	C_6H_5	CH_3	131	66

*PMR spectrum (CF_3COOH, 20°C, hexamethyldisiloxane): 2.30 (s, 6-CH_3), 2.55 (s, 2-CH_3), and 7.15-7.18 ppm (m, phenyl protons).

Rostov State University. Scientific-Research Institute of Physical and Organic Chemistry, Rostov-on-Don 344006. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 5, p. 704, May, 1977. Original article submitted December 7, 1976.