Phenyl isothiocyanate reacts by the same mechanism to form the 3-phenyl-2-thiono-4-oxo-1,2,3,4-tetrahydroquinazoline.

The reaction of isatoic anhydride with a Schiff's base is found to be analogous to the isocyanate reaction. The nucleophilic nitrogen of benzalaniline attacks the number four carbon atom of isatoic anhydride with subsequent loss of CO_2 and ring closure to form 2,3-diphenyl-4-oxo-1,2,3,4-tetrahydroquinazoline. This product was also prepared by the alternate synthesis through *o*-aminobenzanilide as shown below. The physical properties and infrared spectra of the products from the two syntheses were found to be identical (Figure 3).

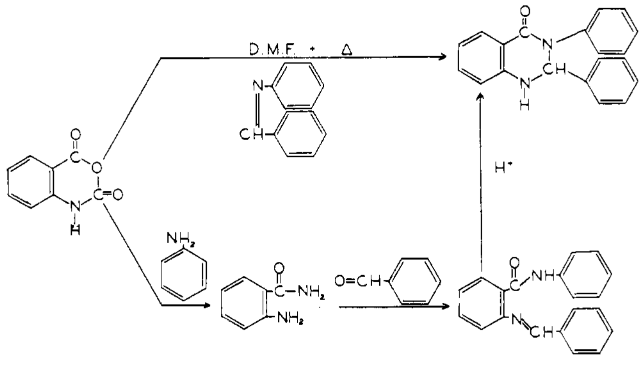

Figure 3. Synthesis of 2,3-diphenyl-4-oxo-1,2,3,4-tetrahydroquinazoline

The established reactions of isatoic anhydride with amines, alcohols, and mercaptans have been extended to enlarge the number of derivatives of isatoic anhydride to facilitate the use of the anhydride as a reagent in organic qualitative analysis or the identification of common nucleophiles. All compounds of new composition appear in Table I.

ACKNOWLEDGMENT

The synthesis of new compounds was conducted by a team of undergraduates, Carol D. Bond, Barbara M. Gattiker, F H. Genter, F.D. Vastine, and C.G. Wade.

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Nitration of Cyclic Sulfites

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IN CONNECTION with another study it became necessary to prepare a series of cyclic sulfites, several of which have not been previously reported. The sulfites were prepared from the corresponding diols by treatment with thionyl chloride, using standard methods (*2*). The sulfites and their physical properties are listed in Table I. The diols were all known compounds.

In the course of working with the sulfites it was found that treatment with common nitrating agents gave the dinitrates of the corresponding diols. In particular, three of the sulfites were cleaved with acetyl nitrate. This represents a new and potentially useful method for the

Table I. Properties of Cyclic Sulfites and Dinitrates

					Analyses							
					Found, %				Calculated, %			
—R—	M.P.[a]	Max. (IR), μ	%, Yield	Sulfites Nitrating Agent	%, C	%, H	%, S	%, N	%, C	%, H	%, S	%, N
$—CH_2CCl_2CCl_2CH_2—$	59–60°	8.17μ	74		17.6	2.09	11.6	...	17.5	1.47	11.7	...
$—CH_2CF_2CF_2CH_2—$	21–22°	...[b]	78		23.5	2.25	15.3	...	23.1	1.92	15.4	...
$—CH_2C(CH_3)_2C(CH_3)_2CH_2—$	31–32°	8.38μ	83		50.3	8.42	16.4	...	50.0	8.32	16.6	...
$—CH_2CCl_2CH_2—$	62–63°	8.40μ	65		18.6	2.62	16.4	...	18.8	2.09	16.7	...
$—CH_2CF_2CH_2—$	n_{D}^{25}1.458		51		22.3	2.79	...	...	22.7	2.53	...	...
—*o*-$C_6H_5(CH_2)_2=$	36–37°	8.48μ	57		52.1	4.41	17.8	...	52.1	4.38	17.4	...
$—(C_5H_3)_2C_2=$	190–191°	8.38μ	62		61.8	3.84	13.4	...	62.0	3.46	13.8	...
—[*o*-$C_6H_5(CH_2)_2NO_2$]	132–133°	8.47μ 6.53μ	42	MA[c]	42.2	3.29	13.7	...	41.9	3.09	13.9	...
$—O_2NC_6H_4—C_2—C_6H_4NO_2$	226–228°	8.27μ	48	AN[c]	44.7	2.07	...	8.55	44.7	1.87	...	8.68
$—C_6H_{10}(CH_2)_2=$	n_{D}^{25}1.4927	8.49μ	...		50.1	7.18	...	...	50.5	7.42	16.9	...
$—CH_2CCl_2CCl_2CH_2—$	70–71°	6.02μ	81	AN[c]	15.4	1.27	...	8.53	15.1	1.25	...	8.78
$—CH_2CF_2CF_2CH_2—$	n_{D}^{25}1.3930	6.00μ	58	MA[c]	19.5	1.64	...	...	19.1	1.58	...	...
$—CH_2C(CH_3)_2C(CH_3)_2CH_2—$	95–96°	6.12μ	66	AN[c]	40.7	6.68	...	11.9	40.6	6.78	...	11.8
$—CH_2CCl_2CH_2—$	n_{D}^{25}1.4766	6.04μ	73	AN[c]	15.8	1.37	...	11.8	15.3	1.70	...	11.8
$—CH_2CF_2CH_2—$	n_{D}^{25}1.4016	6.03μ	52	MA[c]	18.4	1.98	...	...	17.8	1.98	...	...

[a] The melting points are corrected. [b] Sulfite band obscured by CF absorption. [c] MA = Mixed acid; AN = Acetyl nitrate.

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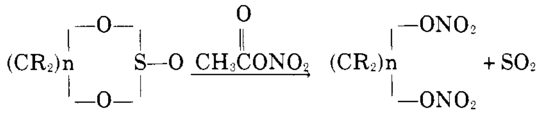
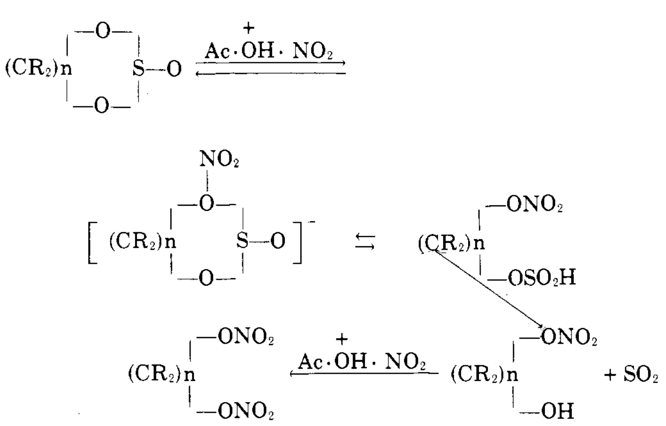
preparation of dinitrates under comparatively mild conditions.



Sulfites are known to hydrolyze under acidic conditions with the cyclic class reacting more slowly (*1, 3, 4*) in acid catalyzed hydrolysis. A possible mechanism based on this hypothesis is shown below.



Several of the dinitrates were new compounds; their properties are listed in Table I. The identity of these compounds was checked in each case by comparison with a sample obtained by nitration of the starting diol with nitric acid-sulfuric acid.

Treatment of the two sulfites containing benzenoid rings with nitrating agents gave only the corresponding nitrosulfites. The sulfite linkage in these compounds resisted cleavage even when a large excess of nitrating agent and longer reaction times were employed.

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Esters of Naphthalenedicarboxylic Acids

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The melting points or boiling points of a number of esters of four positional isomers of naphthalenedicarboxylic acid (1,6-; 2,3-; 2,6-; and 2,7-) are reported. For an ester of a given alcohol, the 2,6- isomer invariably has the higher melting point. With the exception of the dimethyl esters, the order of decreasing melting points for the esters of a given alcohol is 2,6 > 2,7 > 2,3 > 1,6.

A NUMBER OF ESTERS of four positional isomers of naphthalenedicarboxylic acid (1,6-; 2,3-; 2,6-; and 2,7-) were investigated. Apparently, only a limited number of these esters have been previously reported in the literature (*1, 5, 6, 7*), perhaps because of the relative difficulty involved in obtaining the dicarboxylic acids (*1*). In the past few years, however, preparative methods have become available which make possible the oxidation of dimethylnaphthalenes directly to the corresponding dicarboxylic acids (*4, 7, 8*).

EXPERIMENTAL

The esters were prepared from the corresponding diacid chlorides (Method A), by the direct esterification of the dicarboxylic acids (Method B), or by the acid-catalyzed alcoholysis of the 2,3-dicarboxylic acid anhydride (Method C).

The acid chlorides listed in Table I were prepared by the action of an excess of thionyl chloride upon the solid dicarboxylic acids when a small amount of dimethylformamide was added to the reaction mixture (*2, 3*). Similar treatment of the 2,3-dicarboxylic acid, however, resulted in the isolation of the monomeric anhydride (*9*).

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