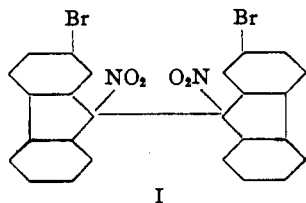


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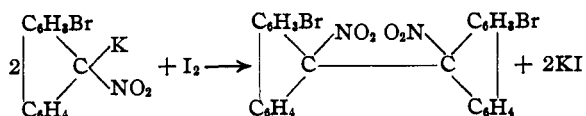
2-Bromo-9-Nitrofluorene and 1,1-Dinitro-3,3'-dibromo-bifluorenyl

BY C. DALE¹ AND R. L. SHRINER

Recently a compound melting at 170° was described as being 2-bromo-9-nitrofluorene.² It is very unusual for a normal nitro compound to melt higher than its aci-form (m. p. 132°). Moreover, the compound decomposes on heating and liberates oxides of nitrogen and forms 2-bromofluorenone. The alkali insolubility of the compound and failure to form a salt of the aci-form, even with potassium methoxide, indicate that it is probably the bimolecular oxidation product with the structure (I) analogous to the product obtained by Nenitzescu³ in his study of aci-9-nitrofluorene.



A reëxamination of this compound has shown that it may be obtained in small amounts by boiling an alcoholic solution of the aci-form, or in good yields by treating the potassium salt of 2-bromo-9-nitrofluorene with one mole of iodine. The product of this reaction is a yellow powder



which shrinks and decomposes over a considerable range of temperature (130–140°). By careful fractionation from methyl acetate, colorless crystals of the compound were obtained. They melted at 172.5° (175° corr.).

The analyses for bromine and nitrogen previously given² are correct, but, of course, would check very closely the theoretical values for either the monomolecular or bimolecular structure. A combustion gave the following results: Calcd. for $\text{C}_{13}\text{H}_9\text{O}_2\text{NBr}$: C, 53.81; H, 2.76. Calcd. for

$\text{C}_{26}\text{H}_{14}\text{O}_4\text{N}_2\text{Br}_2$: C, 54.00; H, 2.42. Found: C, 54.04; H, 2.51.

The molecular weight was determined by the micro boiling point method, using acetone and benzene as solvents. In acetone a molecular weight of 593 was obtained, and in benzene a value of 554 resulted. The calculated value for I, $\text{C}_{26}\text{H}_{14}\text{O}_4\text{N}_2\text{Br}_2$, is 578, whereas the molecular weight of $\text{C}_{13}\text{H}_9\text{BrNO}_2$ is 290. It is evident that the compound is the bimolecular oxidation product with the structure I.

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p-Phenylphenacyl Esters of Organic Acids

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During the course of a research the following *p*-phenylphenacyl esters were prepared which do not appear in the literature. Since the melting points of these may be an aid in the identification of acids they are of value.

They were prepared according to the method of Drake and co-workers¹ and were recrystallized to constant melting point. All melting points are

p-PHENYLPHENACYL ESTERS

Acid	M. p., °C.	Halogen, %	
		Calcd.	Found
<i>m</i> -Bromobenzoic	155 ^a	20.25	20.13
<i>p</i> -Bromobenzoic	160	20.25	20.16
<i>o</i> -Chlorobenzoic	123	10.12	10.09
<i>m</i> -Chlorobenzoic	154 ^b	10.12	10.18
<i>p</i> -Chlorobenzoic	160	10.12	10.19
<i>o</i> -Iodobenzoic	143	28.74	28.38
<i>m</i> -Iodobenzoic	147	28.74	29.21
<i>p</i> -Iodobenzoic	171 (Closed tube)	28.74	28.20
		Nitrogen, %	
<i>o</i> -Nitrobenzoic	140	3.87	3.70
<i>m</i> -Nitrobenzoic	153	3.87	3.95
<i>o</i> -Nitrocinnamic	146	3.61	3.72
<i>m</i> -Nitrocinnamic	193 ^c	3.61	3.66
<i>p</i> -Nitrocinnamic	192	3.61	3.94
<i>p</i> -Cyanobenzoic	165	4.10	4.26
Diphenylacetic	111	C, 82.75	82.98
		H, 5.41	5.66

^a Mixed m. p. with *m*-bromobenzoic acid 127–128°.^b Mixed m. p. with *m*-chlorobenzoic acid 130–132°.^c Mixed m. p. with *m*-nitrocinnamic acid 180–183°.

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(2) Thurston and Shriner, *THIS JOURNAL*, **57**, 2163 (1935).(3) Nenitzescu, *Ber.*, **63**, 2669 (1929); **63**, 2484 (1930).(1) Drake and co-workers, *THIS JOURNAL*, **52**, 3715 (1930); *ibid.*, **54**, 2059 (1932).