solution and naphthas using sodium plumbite and sulfur.[7] Whether the color play in both cases is due to the same original compound is not proved by these experiments.

An attempt was made to use the titration with iodine in benzene represented by the equation $Pb(SR)_2 + I_2 \longrightarrow PbI_2 + (SR)_2$ as an analytical method, but this was not very successful. The reaction was rapid at first, slow after half of the iodine had been absorbed, and toward the end very slow. The benzene used was freshly distilled from sodium. The lead iodide formed was filtered into a Gooch crucible, washed, dried and weighed. The results are given in Table II.

Table II

Results of Analyses

$Pb(SR)_2$	G.	Mg. of iodine Calcd.	Abs.	%	PbI_2 Calcd.	G.	%
Ethyl	0.3664	282.4	272.2	96.39	0.5129	0.5058	98.61
n-Amyl	.2558	157.1	154.5	98.34	.2854	.2750	96.36
Benzyl	.3788	212.1	178.5	84.16	.3853	.3500	90.83

Summary

Lead mercaptides have been prepared and analyzed and some of their properties noted.

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[Contribution from the Chemical Laboratory of the University of Washington]

THE REACTION OF PHTHALYL CHLORIDE WITH AMIDES

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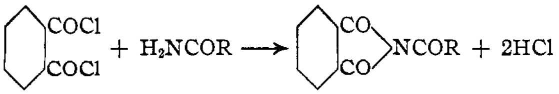
Acetylphthalimide[1] and benzoylphthalimide[2] are the only examples of substitution of the imino hydrogen of phthalimide by acid radicals. The acetyl derivative was prepared by the action of acetic anhydride on phthalimide, also by dehydration of acetylphthalamic acid; the benzoyl derivative was prepared by the action of benzoyl chloride on phthalimide in pyridine solution, also by dehydration of benzoylphthalamic acid.

Because compounds of this type were required in another study, we have investigated different methods for their preparation, and have found that phthalyl chloride reacts normally with amides



giving good yields[3] which were free from other products, especially such as

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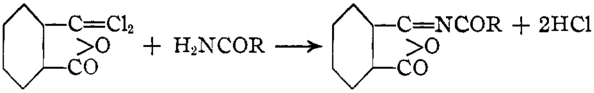
[7] G. L. Wendt and S. H. Diggs, *Ind. Eng. Chem.*, **16**, 1113 (1924); A. E. Wood, A. Lowy and W. F. Faragher, *ibid.*, **16**, 1116 (1924).

[1] Aschan, *Ber.*, **19**, 1400 (1885).

[2] Titherley and Hicks, *J. Chem. Soc.*, **89**, 708 (1906). For a general discussion of methods of acylation of amides, see Titherley, *ibid.*, **85**, 1679 (1904).

[3] Titherly concluded that the action of an acyl chloride on amides is "a poor method, applicable only to the primary amides of the aliphatic series."

might be expected if the isomeric form of phthalyl chloride reacts in accordance with the equation



When equivalents of the phthalyl chloride and the amide were heated, either without a solvent or in toluene until no more hydrogen chloride was evolved, the products could be purified by one crystallization. This method was invariably successful but other methods failed.

For example, *p*-tolylsulfonylphthalimide was easily prepared by this method, but could not be prepared by any of the following methods: heating phthalic anhydride and *p*-toluenesulfonamide, heating potassium phthalimide with *p*-toluenesulfonyl chloride or heating phthalimide with *p*-toluenesulfonyl chloride in pyridine. Also, heating benzenesulfonamide and phthalic acid to 230° failed to give benzenesulfonylphthalimide, easily obtainable from phthalyl chloride and benzenesulfonamide.

Table I

Preparational Data

Crystallized from Toluene

Amide used	Formula A = $C_8H_4O_2N$	Crystal form	M. p., °C.	Calcd. S	Found, S
Acetamide	$ACOCH_3$	Octahedra	135–136	...	...
Benzamide	$ACOC_6H_5$	Prisms	168	...	...
Benzene sulfon-	$ASO_2C_6H_5$	Needles	205	11.12	10.62
o-Tolyl sulfon-	$ASO_2C_6H_4CH_3$	Prisms	182	10.59	10.39
p-Tolyl sulfon-	$ASO_2C_6H_4CH_3$	Prisms	231	10.59	10.36
p-Tolyl-*m*-nitro-sulfon-	$ASO_2C_6H_3CH_3NO_2$	Needles	247	9.26	8.95
Urea[a]	$\overline{C_6H_4CONHCONHCO}$	Needles	188–190	...	...
2,5-Dichloro-aniline	$AC_6H_3Cl_2$[b]	Hair-like Needles	185–190	Cl, 24.28	Cl, 24.68

[a] Piutti, *Ann.*, **214**, 23 (1882), prepared this compound from phthaluric acid.

[b] Crystallized from benzene.

Summary

The imino hydrogen of phthalimide is readily substituted by acid radicals when phthalyl chloride reacts with amides.

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