rate to give the free radicals necessary for initiation of polymerization. Apparently neither of these conditions produces a large concentration of radicals, for only easily polymerizable monomers seem to give polymers. It is of course known[3] that aryldiazonium salts initiate polymerizations of a variety of monomers in alkaline media, but they have apparently not been used for this purpose primarily in acid media.[4]

Experimental

Each of the polymerizations recorded in Table I was accomplished by either shaking (28 and 30° runs) or tumbling (0° and 50° runs) the indicated reaction mixture for the designated period, preliminary experiments having shown no differences in the products from these operations.

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(3) J. M. Willis, G. Alliger, B. L. Johnson and W. M. Otto, Lecture before Division of Rubber Chemistry, American Chemical Society Meeting, Buffalo, New York, October 29, 1952.

(4) After this note had been accepted for publication W. Cooper published a note in *Chemistry and Industry*, No. 17, p. 407, April 25, 1953, describing the use of *p*-nitrobenzenediazonium *p*-chlorobenzenesulfonate as an initiator for the polymerization of methyl acrylate and acrylonitrile in 2.5 *N* hydrochloric acid solution.

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N-Acetylimesatins and Related Compounds

BY FEDERICO PARISI

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In the course of researches carried out some years ago at the University of Fribourg (Switzerland) on the constitution of the products of the reaction of indigo with benzoyl chloride,[1] it was supposed that imesatins were obtained by the degradation of the products of benzoylation with aromatic amines.

This supposition has since been found to be erroneous, although the possibility of the formation of an imesatin by the treatment of Dessoulavy's compound with ammonia is still being studied.

This note will serve to record the preparation of several N-acetylimesatins and of two derivatives of N-chloroacetylisatin.

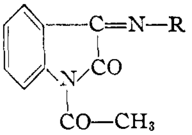
The N-acetylimesatins can be prepared by the action of the corresponding amines on N-acetylisatin (prepared according to J. Büchi[2]) in absolute alcohol without the use of catalysts.

For example: 0.01 mole of N-acetylisatin, 0.02 mole of amine and 30 ml. of absolute alcohol are heated under reflux on the water-bath for an hour, cooled and allowed to stand in the refrigerator to complete the separation of the solid. The product is collected, washed with cold alcohol and then with ether and recrystallized from an appropriate solvent (often glacial acetic acid). It may be necessary to allow the liquid to stand for some days in the refrigerator in order to complete the crystallization. In Table I are given the properties of the substances prepared.

Acetylization of imesatins with acetic anhydride does not produce the corresponding N-acetylimesatins but yields resinous substances.

Derivatives of N-chloroacetylisatin have also been prepared using aniline according to Huntress.[3] In absolute alcohol as for N-acetylimesatins, N-chloroacetylphenylimesatin (I) was obtained. After crystallization from acetic acid it has m.p. 146°.

TABLE I

N-ACETYLIMESATINS 

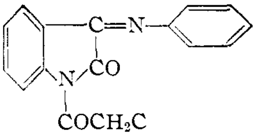
R	Crystallized from	M.p., °C.	Nitrogen, % Calcd.	Nitrogen, % Found
C_6H_5	AcOH	177–178	10.61	10.59
1,2-$C_6H_4CH_3$	AcOH	185–186	10.07	10.13
1,3-$C_6H_4CH_3$	AcOH	169–170	10.07	9.91
1,4-$C_6H_4CH_3$	AcOH	176–177	10.07	10.07
1,2-C_6H_4OH	*i*-Pr_2O	169–170	10.00	9.89
1,4-C_6H_4OH	$Ph{\cdot}CH_3$	194–195	10.00	9.83
1,4-C_6H_4COOH	AcOH	224 (dec.)	9.09	8.94
1-$C_{10}H_7$	AcOH	170–171	8.92	9.01
2-$C_{10}H_7$	AcOH	189–190	8.92	9.03
$C_5H_5N_2$[a]	AcOEt	169–170	20.00	19.82
C_3N_2NS[b]	AcOEt	184–185	15.50	15.41

[a] 4-Methylpyrimidyl. [b] 2-Thiazolyl.

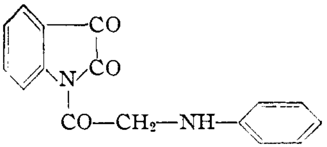
Anal. Calcd. for $C_{16}H_{11}N_2O_2Cl$: N, 9.38; Cl, 11.88. Found: N, 9.57; Cl, 11.71.

On the other hand, 0.01 mole of chloroacetylisatin refluxed with 0.02 mole of aniline in 10 ml. of pyridine for five minutes yields after cooling and diluting with an equal volume of water, half a volume of acetic acid and 8 ml. of concentrated hydrochloric acid (without passing the change point of congo red) a resinous precipitate. When stirred for some time this precipitate redissolves in the liquid leaving only a small residue. The filtrate from this, made alkaline with ammonia, gives a yellow precipitate of phenylglycylisatin (II) soluble in mineral acids, in concentrated alkalies and in ethyl alcohol. Crystallized from acetic acid, it has m.p. 197°.

Anal. Calcd. for $C_{16}H_{12}N_2O_3$: N, 9.99; Cl, nil. Found: N, 10.16; Cl, absent.



I



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(1) H. de Deisbach and coll., *Helv. Chim. Acta*, **16**, 148 (1933); **17**, 113 (1934); **19**, 1213 (1936); **20**, 132 (1937); **23**, 469 (1940); **24**, 158 (1941); **26**, 1869 (1943); **31**, 724 (1948); **32**, 1214 (1949).

(2) J. Büchi and H. Humi, *Helv. Chim. Acta*, **32**, 1806 (1949).

(3) E. H. Huntress and J. Bornstein, THIS JOURNAL, **71**, 745 (1949).

Polarography of Tripositive Antimony and Arsenic. Cathodic Reduction of Antimonous in Strong Hydrochloric Acid and Anodic Oxidation of Arsenite and Stibnite in Strong Sodium Hydroxide

BY G. P. HAIGHT, JR.[1]

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The polarography of arsenic and antimony in acid solutions has been investigated by Lingane,[2] Kolthoff and Probst[3] and Bambach[4] who have shown that the tripositive state of each element is generally reducible while the pentavalent states are reduced only in very strong hydrochloric acid. Waves in 1.5 *M* HCl and in 1 *M* HCl plus 1 *M* tartaric acid have been found suitable for simultane-

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(1) Chemistry Department, University of Kansas, Lawrence, Kansas.

(2) J. J. Lingane, *Ind. Eng. Chem., Anal. Ed.*, **15**, 583 (1943).

(3) I. M. Kolthoff and R. L. Probst, *Anal. Chem.*, **21**, 753 (1949).

(4) K. Bambach, *Ind. Eng. Chem., Anal. Ed.*, **14**, 265 (1942).