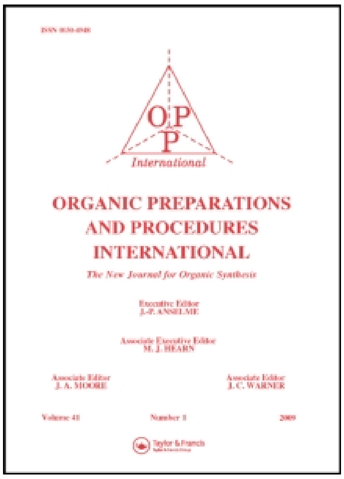

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3- AND 4-(7-BENZ[a]ANTHRACENYL)BENZAMIDES AND CYANOBENZENES AND 3- AND 4-(9-ANTHRACENYL)BENZAMIDES AND CYANOBENZENES

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3- AND 4-(7-BENZ[a]ANTHRACENYL)BENZAMIDES AND CYANOBENZENES AND 3- AND 4-(9-ANTHRACENYL)BENZAMIDES AND CYANOBENZENES

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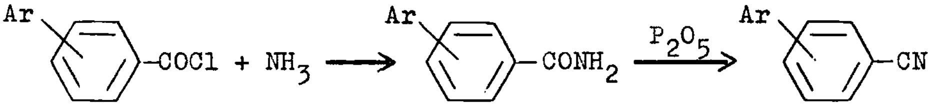

1-4 5-6 9-12

Ar = 9-Anthracenyl for 6,8,9,11

Ar = 7-Benz[a]anthracenyl for 5,7,10,12

The title compounds (all gave satisfactory elemental analyses) were prepared from their respective amides by dehydration with P_2O_5. The amides were prepared from the carboxylic acids[2] *via* their respective acid chlorides[3] by treatment with liquid ammonia in tetrahydrofuran (THF). The dehydration reaction was found to be a better synthetic procedure for the preparation of the aromatic nitriles than the cyclodehydration of the corresponding cyanoketones with conc. H_2SO_4.[4]

EXPERIMENTAL[5]

General Procedure for Preparation of Amides

The acid chloride (see Table I for the amounts used) was dissolved in 400 ml of THF (for 5, 7 and 8; 6 required 650 ml of THF), a small quantity of liquid ammonia (HOOD!) was added and the mixture was stirred magnetically as the excess liquid ammonia was allowed to evaporate. The mixture was concentrated, the resultant viscous solid was suspended in 75 ml of 95% ethanol, collected by filtration, washed with water (300 ml) and dried in a vacuum oven to constant weight. The crude yields, the melting points of the purified products and the solvents used for recrystallization are given in Table I.

General Procedure for Preparation of Nitriles

To a solution of the amide (see Table II for amounts) in ~1 l. of refluxing toluene, contained in a 2-l. flask equipped with a mechanical stirrer, was added 4-5 times as much (on a molar basis) phosphorus pentoxide, and the mixture was refluxed for 24 hr. (After 1 hr. the bottom of the flask became coated with a viscous oil.) The mixture was cooled to room temperature, the solvent was decanted from the oil and was concentrated to give a semi-solid. This material was dissolved in a mixture of 100 ml of THF and 100 ml of 10% sodium hydroxide, the solution was poured into the reaction flask which contained the oil and was stirred at room temperature for 1 hr. The THF layer was separated, the aqueous layer was saturated with sodium chloride and extracted twice with 50 ml of THF. The THF layers were combined and were washed succes-

TABLE I

RCOCl (from X g. of acid)	$RCONH_2$	mp. (solvent)	Crude Yield
2, 12 g.	5 4-(7-Benz[a]anthracenyl) benzamide	289-300° white crystals ($CHCl_3$:95% EtOH, 7:3	89%
1, 15 g.	7 3-(7-Benz[a]anthracenyl) benzamide	135-7° white crystals (95% EtOH, charcoal)	87%
4, 13 g.	6 4-(9-Anthracenyl) benzamide	270-1° white needles (THF:95% EtOH, 7:3)	86%
3, 15 g.	8 3-(9-anthracenyl) benzamide	190-200° yellow solid (95% EtOH)	—

TABLE II

$RCONH_2$ X g.	RC≡N	mp. (solvent)	Yield
5, 9 g.	9	197-8° lit.[4] 196-8° white crystals (95% EtOH)	64%
7, 9 g.	11	153-5° white powder (ØH:EtOH, 1:3)	65%
6, 9 g.	10	134-5° lit.[6] 125-6° yellow needles (95% EtOH)	66%
8, 10 g.	12	129-30° yellow plates ($CHCl_3$:95% EtOH, 1:4)	61%

sively with 100 ml of water, 100 ml of 10% sodium bicarbonate solution and twice with 100 ml of water. The separated THF layers were dried over sodium sulfate, were filtered and were concentrated to give a viscous oil. The oil was dissolved in 50 ml of ethyl acetate, and cooled overnight to yield 1.5 g of unreacted amide which was separated by filtration. Concentration of the filtrate produced an oil which was dissolved in 30 ml of benzene and chromatographed on a silica gel column using benzene (300 ml) as the eluent. Concentration of the benzene gave an oil which crystallized when 25 ml of ethanol (95%) was added (9, 10, 12; 11 crystallized after the oil was dissolved in benzene-heptane (1:3)). The yields and mp. of purified products and the solvents used for recrystallization are given in Table II.

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1. a) Present address: Northeast Louisiana State College, Department of Chemistry, Monroe, Louisiana 71201;
   b) Abstracted from the Ph.D. Thesis of M.P.R., Nov. 1969;
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5. The melting points of all compounds were obtained with a Fisher-Johns melting point block and are corrected. Analyses were performed by Galbraith Labs., Garden City, Michigan; and on a departmental F and M Scientific Corp., Model 185, C, H, and N analyzer. The chromatography

columns were 1½ in. in diameter and 11 in. in length, and were wet packed with Baker's Silica Gel powder, "Suitable for Chromatographic Use," with benzene, and were eluted with benzene.

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