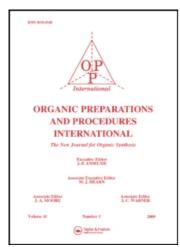
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# 3- AND 4-(7-BENZ[A]ANTHRACENYL)PHENYL-2-IMIDAZOLINES, 3-AND 4-(9-ANTHRACENYL)PHENYL-2-IMIDAZOLINES, AND THEIR HYDROCHLORIDE SALTS

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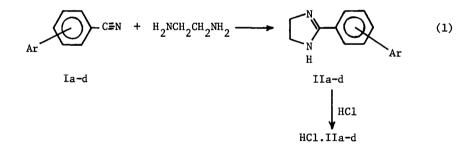
### ORGANIC PREPARATIONS AND PROCEDURES INT. 7(3). 145-148 (1975)

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AND THEIR HYDROCHLORIDE SALTS.

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The four 2-imidazolines described in eq. 1 were prepared  $^1$  by the reaction of ethylenediamine with the cyanobenzene derivatives.  $^2$ 



- a) 3-(7-Benz[a]anthracenyl)-
- b) 4-(7-Benz[a]anthraceny1)-
- c) 3-(9-Anthraceny1)-
- d) 4-(9-Anthracenyl)-

2-Imidazolines gave the resonance-stabilized hydrochloride salts (HCl.IIa-d) by the addition of an ethyl ether solution saturated with anhydrous hydrogen chloride to an anhydrous tetrahydrofuran solution containing the respective 2-imidazolines.

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### EXPERIMENTAL<sup>4</sup>

General Procedure For Preparation of 2-Imidazolines.- Into a 100 ml, one-necked, round-bottomed flask, equipped with a condenser and a magnetic stirrer, was placed the substituted cyanobenzene and the ethylenediamine, and the reaction allowed to reflux for 5-6 hrs. The mixture was cooled overnight, the solid filtered and washed with 100 ml of water, and the product dried at 35° (0.3 mm) for 4-5 hrs. Recrystallization afforded the product as a solid in all cases. NMR for IIa,b (DMSO)  $\delta$  5.25-4.0 (m, 15, ArH), 3.1 (s, 1, -NH), 2.0 (s, 4, (CH2)<sub>2</sub>); for IIc,d (CDCl<sub>3</sub>) 4.7-4.0 (m, 13, ArH), 2.5 (s, 1, -NH), 2.0 (s, 4, (CH2)<sub>2</sub>).

Table I
Preparation of 2-Imidazolines

Cyanobenzene amount		Ethylenediamine amount	Yield		mp,	
No.	g (moles)	m1		crystals	mp, °C	
Ia	5.0(0.015)	50 <sup>a</sup>	4.1(11.0), 73.4 <sup>b</sup>	white	227-229	
Ιb	4.0(0.012)	30	3.7(9.9), 82.7 <sup>c</sup>	yellow- scaly	221.5-223	
Ic	5.0(0.018)	50 <sup>d</sup>	4.2(13.0), 72.4 <sup>e</sup>	light- yellow	241-223	
Id	5.0(0.018)	40	4.8(15.0), 82.7 <sup>f</sup>	light- yellow needles	309 dec.	

<sup>&</sup>lt;sup>a</sup>Solvent removed under reduced pressure and the resulting brown semisolid crystallized from 25 ml of methanol.

bFour recrystallizations from 95% ethanol-ethyl acetate (1:9), and one treatment with charcoal.

<sup>&</sup>lt;sup>C</sup>Five recrystallizations from chloroform-ethyl acetate (1:1).

<sup>&</sup>lt;sup>d</sup>Solvent removed under reduced pressure and the resulting brown semisolid crystallized from 25 ml of 95% ethanol.

<sup>&</sup>lt;sup>e</sup>Four recrystallizations from chloroform-95% ethanol (3:4), and one treatment with charcoal.

 $<sup>^{</sup>m f}$  Five recrystallizations from chloroform-95% ethanol (3:7).

#### PHENYL-2-IMIDAZOLINES AND THEIR HYDROCHLORIDE SALTS

General Procedure For Preparation of 2-Imidazoline Hydrochlorides.— Into a 250 ml, one-necked, round-bottomed flask, equipped with a condenser and a magnetic stirrer, was placed 2.5 g of the respective 2-imidazoline dissolved in 100 ml of hot THF and 50 ml of anhydrous ether saturated with hydrogen chloride (60 ml in the case of IIa and IIc) was added. The mixture was stirred magnetically at room temperature for 15 min, then concentrated. IIb and IId gave a white solid at this point, whereas IIa and IIc gave an oil which was dissolved in 25 ml of warm absolute ethanol and crystallized when 50 ml of ethyl acetate was added. Five recrystallizations afforded a solid product in all cases.

Table II

Preparation of 2-Imidazoline Hydrochlorides

No.	Recrystalliza Solvent	ations Ratio	Yield g (mmole), % crystals	mp, °C	
IIa	95% EtOH	-	2.5(6.1), 91.0 white flaky	201-204	
IIb	95% EtOH-EtOAc	8:2	2.2(5.4), 88.5 white powder	325 dec.	
IIc	EtOH-EtOAc	3:1	2.3(6.4), 95.5 yellow	314 dec.	
IId	95% EtOH-CHC1 <sub>3</sub>	3:1	2.4(6.6), 98.5 light yellow needles	333 dec.	

Table III

Elemental Analysis For 2-Imidazolines and Their Hydrochloride Salts

	_	%Calcd.			%Found				
C'mpd.	Formula	С	H	N	C1	C	Н	N	C1
IIa	C <sub>27</sub> H <sub>20</sub> N <sub>2</sub>	87.05	5.42	7.52		86.91	5.42	7.35	
IIb	$^{\mathrm{C}}^{}_{27}^{\mathrm{H}}^{}_{20}^{\mathrm{N}}^{}_{2}$	87.05	5.42	7.52		87.20	5.38	7.47	
IIc	$^{\mathrm{C}}^{23}^{\mathrm{H}}_{18}^{\mathrm{N}}_{2}$	85.67	5.64	8.69		85.91	5.86	8.43	
IId	$^{\mathrm{C}}_{23}^{\mathrm{H}}_{18}^{\mathrm{N}}_{2}$	85.67	5.64	8.69		85.54	5.77	8.65	
IIa.HC1	$^{\mathrm{C}}_{27}^{\mathrm{H}}_{21}^{\mathrm{C1N}}_{2}$	79.29	5.19	6.85	8.67	79.37	5.28	6.64	8.87
IIb.HC1	$^{\mathrm{C}}_{27}^{\mathrm{H}}_{21}^{\mathrm{C1N}}_{2}$	79.29	5.19	6.85	8.67	79.35	5.34	6.75	8.74
IIc.HC1	$^{\mathrm{C}}_{23}^{\mathrm{H}}_{19}^{\mathrm{C1N}}_{2}$	76.97	5.35	7.81	9.88	76.69	5.42	7.54	10.18
IId.HC1	C23H19C1N2	76.97	5.35	7.81	9.88	77.13	5.30	7.83	10.10

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- †Present address: Northeast Louisiana State College, Department of Chemistry, Monroe Louisiana 71201; ††Abstracted from the Ph.D. Thesis of M. P. R., Nov. 1969; \*To whom inquiries should be sent.
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- 4. The melting points of all compounds melting below 300° were taken on a Fisher-Johns melting point block and are corrected; those melting above 300° were taken on a Mel-Temp capillary melting point apparatus 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 NMR spectra were recorded on a Varian A-60 spectrophotometer, using 10% deuterated chloroform or DMSO solutions with tetramethylsilane (TMS) as an internal standard.

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