Even-Numbered *n*-Acyl and *n*-Alkyl Ferrocenes

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The Friedel-Crafts acylation with acid chlorides and aluminum chloride was one of the first reactions to be carried out with ferrocene.[2] About a dozen each of *n*-acyl- and *n*-alkyl ferrocenes with various chain lengths have since been prepared.[3-5]

TABLE I

PROPERTIES OF FERROCENES

Substituent	Color	Yield	B.P. or Solvent	M.P.[a] or n_D^{20}	Calcd. C	Calcd. H	Found C	Found H
ACYL SUBSTITUTED								
1-Ethanoyl-[b]	Red	—	Methanol	81–83°	—	—	—	—
1-Butanoyl-	Red	72%	144–146°/1.5 mm.	1.6073	65.6	6.3	65.8	6.2
1-Hexanoyl-	Orange	88%	161–163°/1.5 mm.	1.5843	67.6	7.0	67.4	7.0
1-Octanoyl-	Orange	91%	Methanol	26–27°	69.2	7.7	69.1	7.4
1-Decanoyl-	Red	84%	203–204°/1.6 mm.	1.5513	70.6	8.2	70.8	8.2
1-Dodecanoyl-	Orange	79%	Methanol	36–37°	71.7	8.6	71.7	8.5
1,1′-Dihexanoyl-	Orange	67%	Methanol	38–39°	69.1	7.8	68.8	7.7
ALKYL SUBSTITUTED								
1-Ethyl-[c]	Red	67%	107–108°/5 mm.	1.6011	—	—	—	—
1-Butyl-[c]	Red	7%	180°/3/5 mm.	1.5701	—	—	—	—
1-Hexyl-	Orange	93%	139–40°/1.5 mm.	1.5602	71.1	8.1	71.2	8.1
1-Octyl-	Orange	71%	154–155°/1.0 mm.	1.5490	72.5	8.7	72.6	8.7
1-Decyl-	Orange	90%	183–184°/1.4 mm.	1.5399	73.6	9.2	73.9	9.3
1-Dodecyl-	Yellow	96%	Methanol	35–36°	74.6	9.6	74.6	9.6
1,1′-Dihexyl-	Orange	86%	189°/1.6 mm.	1.5320	74.6	9.6	74.8	9.9

[a] Uncorrected. [b] From A. N. Nesmeyanov, E. G. Perevalova, R. V. Goloonya, and O. A. Nesmeyanova, *Doklady Akad. Nauk S.S.S.R.*, 97, 459 (1954). [c] From Ref. 3.

To complete an even-numbered series of *n*-acyl and *n*-alkyl ferrocenes, eleven new ferrocenes through dodecyl were synthesized in good yields by the methods of Rausch.[5] After purification by either distillation through a 60 × 8-mm. spinning-band column or recrystallization from methanol, the products were red to yellow liquids and low-melting solids. Table I lists the measured properties.

All of the acyl derivatives showed carbonyl infrared absorption at about 6 μ. The monosubstituted ferrocenes showed infrared absorption bands at 9 to 10 μ.[6]

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(2) R. B. Woodward, M. Rosenblum, and M. C. Whiting, *J. Am. Chem. Soc.*, 74, 3458 (1952).

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8-Quinolinol Derivatives of Borinic Acids

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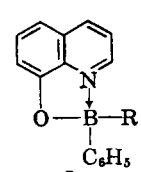
In 1955, Letsinger and Skoog reported that diarylborinic acids react with ethanolamine to form crystalline products.[1] Attempts in this laboratory to prepare such derivatives of several aralkylborinic acids were not uniformly successful, and thus attention was turned to the problem of finding a suitable reagent for making derivatives of these acids. As ethanolamine and 8-quinolinol have in common the critical reactive grouping, HO—C(—)—C(—)—N=, the latter was considered as a possible reagent, and indeed it was found to react with both diaryl- and aralkylborinic acids to form yellow crystalline products (I) which are readily isolated and characterized.[2] Each of the reported compounds (Table I) shows an intense green fluorescence under ultraviolet light.



I

(1) R. L. Letsinger and I. Skoog, *J. Am. Chem. Soc.*, 77, 2491 (1955).

(2) R. Neu [*Z. anal. Chem.*, 142, 335 (1954)], in an article describing the use of diphenylborinic acid as a reagent for identifying certain 5-hydroxyflavones, mentioned that this acid also reacts with 8-quinolinol.