# Synthesis of Dibenzo[*a*,*l*]pyrene

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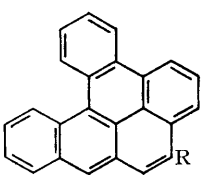
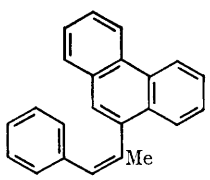
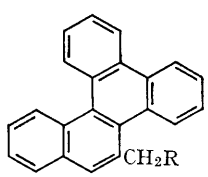
LAIVT-LAMY and BUU-HOÏ have recently reported[1] that the hydrocarbon hitherto regarded as dibenzo[*a*,*l*]pyrene (I; R = H) is in fact dibenzo[*a*,*e*]fluoranthene, produced by a previously unsuspected rearrangement. Authentic dibenzo[*a*,*l*]pyrene has now been synthesised by an unequivocal route and, in agreement with the French authors, its properties are different from those previously recorded.[2]




(I)




(II)




(III)

Photocyclisation[3] of the styrylphenanthrene (II), readily available from 9-acetylphenanthrene and benzylmagnesium bromide, in hexane solution, afforded the methylbenzochrysene derivative (III; R=H), m.p. 164°, $\lambda_{max}$ (EtOH) 274, 285, 294, 320, 332, infl. 345, 385 m$\mu$ (log $\epsilon$, 4·72, 4·81, 4·82, 4·02, 4·05, 3·85, 3·09), converted by standard reactions into the acetic acid (III; R=$CO_2H$), m.p. 188—189°. Cyclisation of the corresponding acid chloride with stannic chloride in benzene solution gave the phenol[4] (I; R=OH) whence dibenzo[*a*,*l*]pyrene was obtained by reduction with zinc dust in a melt of zinc chloride and sodium chloride.[5] After

purification by chromatography on alumina the hydrocarbon formed pale yellow plates, m.p. 163—164°, $\lambda_{max}$ (EtOH) 240, 271, infl. 282, 294, 305, 319, 341, 360, 375, 397, 420 m$\mu$ (log $\epsilon$, 4·54, 4·64, 4·45, 4·54, 4·69, 4·74, 3·85, 4·02, 4·25, 4·27, 3·04) mol. wt. by mass spectrometry) 302. The 1,3,5-trinitrobenzene complex formed crimson needles, m.p. 205—206°. In concentrated sulphuric acid the hydrocarbon gave a transient deep blue colour, rapidly changing to crimson.

Another not wholly unambiguous synthesis of dibenzo[*a*,*l*]pyrene has been reported[6] since the present work was completed. No evidence of structure is presented, but the melting point of the product is very close to that found in the present work.

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[2] E. Clar, *Ber.*, 1930, **63**, 112; E. Clar and D. Stewart, *J. Chem. Soc.*, 1951, 687.
[3] *Cf.* P. Hugelshofer, J. Kalvoda, and K. Schaffner, *Helv. Chim. Acta*, 1960, **43**, 1322.
[4] *Cf.* R. Weitzenböck, *Monatsh.*, 1913, **34**, 193.
[5] E. Clar, "Polycyclic Hydrocarbons", Academic Press, London, 1964, Vol. I, p. 162.
[6] F. A. Vingiello, J. Yanez, and E. J. Greenwood, *Chem. Comm.*, 1966, 375.