Isolation of 3-Guaiacylbenzanthrone from Anthraquinone Pulping Liquors

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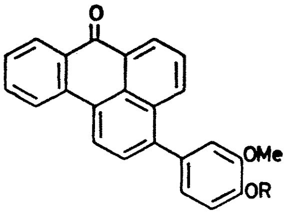
Summary An orange compound isolated from soda-anthraquinone pulping liquors of *Pinus radiata* has been identified as 3-(4-hydroxy-3-methoxyphenyl)-7*H*-benz[*de*]anthracen-7-one (**1a**).

A RECENT report[1] describing the isolation of a substituted benzanthrone of undetermined structure from the black liquors produced by soda-anthraquinone pulping of *Pinus radiata* prompts us to report the structure of a compound, m.p. 198—200 °C which we have isolated from similar liquors by extraction with chloroform and purification by t.l.c. We have identified this as 3-(4-hydroxy-3-methoxyphenyl)-7*H*-benz[*de*]anthracen-7-one (**1a**) from its spectral data and independent syntheses. Its high-resolution mass spectrum showed the molecular ion at *m/e* 352.1100 ($C_{24}H_{16}O_3$). The location of the guaiacyl substituent at the 3-position of the benzanthrone was predicted on the basis of a recently reported related condensation of allyl bromide with 9,10-dihydroxyanthracene in aqueous alkali.[2] Compound (**1a**) could also be obtained by heating anthraquinone with coniferyl alcohol in alkali at 150 °C, using glucose to reduce the anthraquinone to anthrahydroquinone.



(**1**)
a; R = H
b; R = Me

The methyl ether (**1b**), m.p. 224—226 °C, of (**1a**) was synthesised by a mixed Ullman reaction between 3-bromobenzanthrone and 4-bromoveratrole. Compound (**1b**) was identical (mixed m.p., spectra, and polarity) with the methylated derivative of (**1a**).

Isolation of this guaiacyl-substituted benzanthrone provides an explanation of why anthraquinone is only partially recoverable after pulping.

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[1] A. Farringon, P. F. Nelson, and N. Vanderhoek, *Appita*, 1979, **32**(4), 300.
[2] R. J. Deshpande, *Indian J. Chem.*, 1978, **16B**, 389.