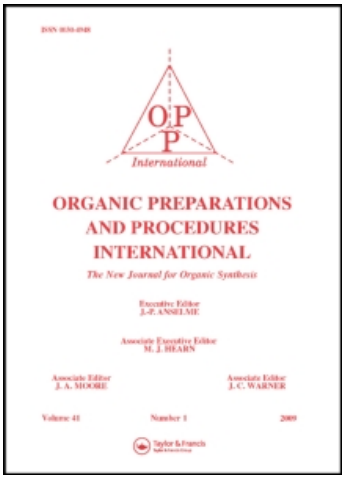

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PREPARATION OF PERDEUTERO 9,10-DIHYDROANTHRACENE

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OPPI BRIEFS

(By J.-P. Anselme, Editor)

PREPARATION OF PERDEUTERO 9,10-DIHYDROANTHRACENE

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(5/9/77)
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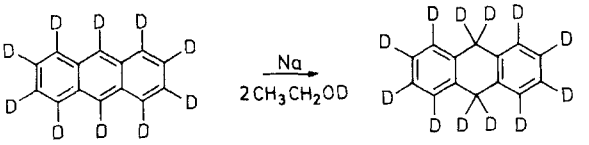
Perdeutero 9,10-dihydroanthracene has been obtained quantitatively from reduction of perdeuteroanthracene with sodium sand[1] and monodeutero ethanol (2 eq.) in xylene.



EXPERIMENTAL[2]

Dry xylene (250 ml) and 4.5 g of sodium were heated to 130° in a 1 ℓ. three-necked flask fitted with a Vibromixer, a reflux condenser and a dropping funnel. As soon as the sodium was finely divided in droplets, the temperature was lowered to 100° and 2.3 g of perdeuteroanthracene was added. The temperature was kept at 100° and 20 ml of monodeutero ethanol was added over a period of 45 min. After cooling, 250 ml water was added to destroy the sodium ethanolate. The xylene layer was dried over $MgSO_4$ and after filtration the xylene was removed with a rotary evaporator. The residue

was essentially perdeutero 9-10-dihydroanthracene containing less than 0.1-0.2% perdeuteroanthracene (glc). The product was chromographed over Merck silica gel 60 (0.063-0.200 mm) with hexane as eluent to give 2.05 g (89%) of perdeutero 9,10-dihydroanthracene, mp 107-108°. The product contained less than 0.2% deutero anthracene. Mass spectra indicated the deuterium incorporation to be 97%. The distribution of isotopic species $C_{10}D_{12}$, $C_{10}HD_{11}$, etc. could not be established because the predominance of D(H) loss from the parent ions in the fragmentation process. The NMR spectrum showed that the amount of ^{1}H on the 9-10 positions is 2.5 times greater than on the benzene ring positions. The highly characteristic IR spectrum (KBr) differing completely from the parent dihydro compounds exhibited peaks at 2283, 2265, 2203, 2078, 2108 cm^{-1} and very weak absorptions between 2800 and 2950 cm^{-1}. Perdeuteroanthracene was obtained from Merck, D incorporation > 98%. Mass spectrometry showed this material to contain $C_{10}H_3D_7$ 0.12%, $C_{10}H_2D_8$ 1.18%. $C_{10}HD_9$ 17.76% and $C_{10}D_{10}$ 80.33%. Monodeutero ethanol Merck had a minimum deuterium incorporation of 99%.

ACKNOWLEDGEMENT.- The authors wish to thank Prof. J. H. van der Waals for a gift of perdeuteroanthracene.

REFERENCES

1. A. Vogel, Practical Organic Chemistry, p. 193, Longmans Green and Co., London, 1956.

2. A AEI 904 mass spectrometer was used. The NMR spectra were recorded on a JEOLCO PS 100 and the IR spectra on a Unicam SP 1200 instrument. GLC analyses were carried out using an OV 17 capilary column., length 20 m, inside diam. 0.4, flow 3 ml/min., temp. 200°, injection temp. 220°, detector temp. 220°, pressure in injection room 0.15 atm., injection piece with septum coil, retention times: acetone = 3 min, 9,10-dihydroanthracene = 7 min., anthracene = 10 min.