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SYNTHESIS OF 1,2'-BIADAMANTANE

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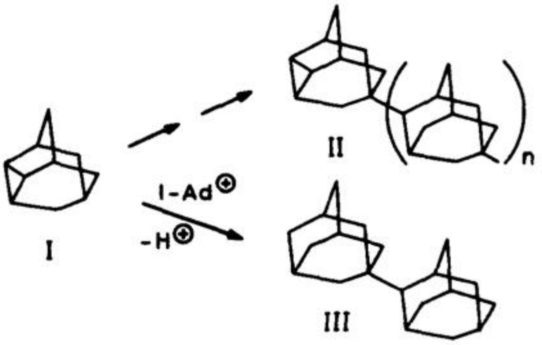
On the aluminium chloride catalysed oligomerization of 2,4-dehydroadamantane (I) containing ca. 1% adamantane, small amounts of a hydrocarbon possessing no cyclopropane ring could be detected in the fraction of the dimeric product (II, n=1).[1] It was assumed that the hydrocarbon might have been the hitherto unknown 1,2'-biadamantane (III) arisen from (I) and 1-adamantyl cation as depicted in Scheme 1.



Scheme 1

In order to confirm the involvement of adamantane in the reaction, varying ratios of (I) and adamantane were treated with aluminium chloride/carbon disulfide.[1] The results are given in Table 1.

I Adamantane	II(n=1) III	(II(n=1) + III II(n > 1) 100 (%)
99/1	5.7	1
50/50	0.5	45
5/95	0.08	99

Table 1

Thus, an increase in the amount of adamantane in the reaction mixture lowers the yield of (II, n=1) relative to (III) *and* the yields of the polymeric products (II, n > 1).

(III) was separated from (II, n=1) by the action of acetic acid at 100 °C for *ca*. 15 hrs. followed by column chromatography (aluminum oxide) of the solvolysed products. Minor amounts of monomeric materials were eventually separated by preparative g.l.c. from (III) which proved to be a crystalline, colourless compound, $C_{20}H_{30}$, m.p. 266°-268 °C (sealed capillary). A prepared sample of 1,1'-biadamantane[2] was shown to be different from (III) by g.l.c. analyses and mixed melting points. A m.s. analysis of (III) gave the molecular ion (m/e=270) whose intensity was *ca*. 15% of the main fragment M-135 (m/e=135) as compared with the *ca*. 90% fragmentation of 1,1'-biadamantane to m/e=135 under the same conditions. The infra-red spectrum of (III) is, like the spectrum of adamantane, essentially featureless. The possibility of the new hydrocarbon being 2,2'-bi-adamantane is excluded because of the latter's much lower melting point (184°-185 °C).[3]

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