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Cyclic Oligophosphonic Anhydrides in Phosphonation of Diacylamides

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Cyclic Oligophosphonic Anhydrides in Phosphonation of Diacylamides

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Diacylamides (10 compounds) were reacted with the system $H_3PO_3 - PCl_3$ in the media of amine hydrochlorides (conditions as in [1], B = Py, Bu_3N) and formed bicyclic dianhydrides 1 in high yields. The reaction failed when none of R was H and when R^1 and R^2 were a part of cycle. Less sterically hindered 1 were opened to monocyclic anhydrides 2 and to acyclic acids 3 using mild acidic hydrolysis.

a: $R^1=R^2=Me$, $R^3=H$; b: $R^1=R^2=C_7H_{11}$, $R^3=H$; c: $R^1=Me$, $R^2=p$ -CH₃C₆H₄, $R^3=H$; d: $R^1=Me$, $R^2=C_{11}H_{23}$, $R^3=H$; e: $R^1=Me$, $R^2=R^3=H$; f: $R^1=p$ -CH₃C₆H₄, $R^2=R^3=H$; g: $R^1=Me$, $R^2=H$, $R^3=Me$; h: $R^1=C_9H_{19}$, $R^2=H$, $R^3=Me$; i: $R^1=R^2=H$, $R^3=Me$; j: $R^1=R^2=H$, $R^3=C_{14}H_{29}$

As a rule, only one of the two possible diastereomers of 2 formed in the hydrolysis (probably having the cis-structure). In two cases cis-isomers (2g,h) were labile, and transformed into the stable trans-2g,h under the conditions of hydrolysis. The compounds 1b,c, 2a,e,f (most likely - cis-isomers), trans-2g,h, cis-2h and 3e,i,j were isolated from the reaction mixtures as pyridine or aniline salts. In addition to ³¹P NMR, the structures of isolated compounds were demonstrated by ¹H NMR and elemental analysis. Both 1 and 2 are stable in alkaline and neutral media. Most of 1, 2 and 3 decompose in hot acidic aqueous solutions with elimination of a half of phosphorus in the form of H₃PO₃.

References

[1] I.S. Alferiev and S.Yu. Bobkov, Z. Naturforsch., 47b, 1213 (1992).