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Nucleosides, Nucleotides and Nucleic Acids

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713597286>

Amination of Ethyl 5-Amino-1-Qlycofuranosylimidazole-4-Carboxylates Using Trimethylaluminium

A. Grouiller^a; R. W. Humble^b; G. Iveson^b; G. Mackenzie^b; B. Najib^a; H. Pacheco^a; G. Shaw^c

^a Institute National des Science Appliquees de Lyon, ^b Humberside College of Higher Education, Hull, U.K. ^c University of Bradford, Bradford, U.K.

To cite this Article Grouiller, A. , Humble, R. W. , Iveson, G. , Mackenzie, G. , Najib, B. , Pacheco, H. and Shaw, G.(1987) 'Amination of Ethyl 5-Amino-1-Qlycofuranosylimidazole-4-Carboxylates Using Trimethylaluminium', *Nucleosides, Nucleotides and Nucleic Acids*, 6: 1, 399 – 400

To link to this Article: DOI: 10.1080/07328318708056236

URL: <http://dx.doi.org/10.1080/07328318708056236>

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AMINATION OF ETHYL 5-AMINO-1-GLYCOFURANOSYLIMIDAZOLE-4-CARBOXYLATES
USING TRIMETHYLALUMINIUM

A. Grouiller^a, R. W. Humble^b, G. Iveson^b, G. Mackenzie^b, B. Najib^a,
H. Pacheco^a and G. Shaw^c.

^aInstitute National des Science Appliquees de Lyon, Bat. 406;
^bHumberside College of Higher Education, Cottingham Road, Hull, HU6 7RT,
U.K.; ^cUniversity of Bradford, Bradford, BD7 1DP, U.K.

Abstract: Ethyl 5-amino-1-β-D-ribofuranosyl- and arabinofuranosyl
imidazole-4-carboxylates were aminated by ammonia, primary and secondary
amines to their corresponding 4-carboxamides using trimethylaluminium.

We have earlier recorded^{1,2} the synthesis of a series of ethyl
5-amino-1-glycofuranosylimidazole-4-carboxylates as precursors to inter-
mediates (and their derivatives/analogues) involved in the de novo bio-
synthesis of purines. Conversion of these imidazole nucleoside esters to
their corresponding carboxamides has been achieved via acid chloride³ or
active ester intermediates^{4,5}. Alternatively, direct aminolysis has been
achieved^{2,6} by use of high temperatures (100°C to 120°C) and long reaction
times (2 to 4 days). Such routes have usually resulted in low yields with
some nucleoside decomposition. We now record a milder and more convenient
method of effecting similar conversions, such as those identified in FIG,
using trimethylaluminium⁷. Aminations were achieved in almost quantitative
yields whether using ammonia or primary or secondary amines. In a typical
experiment, trimethylaluminium (2 molar equivalents) in hexane was slowly
added at room temperature to the amine (1.2 molar equivalents) or ammonia
(several fold excess) in dry methylene chloride under nitrogen. The mix-
ture was stirred at room temperature for 15 minutes and the ester nucleo-
side (1 molar equivalent) added. The reaction mixture was refluxed under
nitrogen (15 to 24 h) until TLC indicated that reaction was complete. The
reaction was cooled, carefully quenched with water and extracted with
methylene chloride. The organic layer was washed with sodium hydrogen
carbonate solution, dried (magnesium sulphate) and evaporated to give the
product which was further purified using silica gel column chromatography.

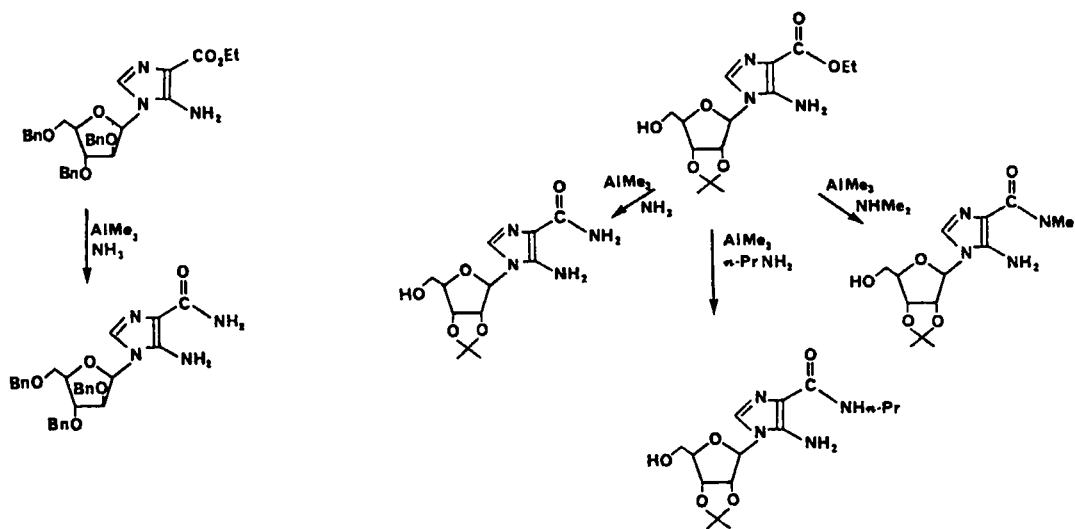


FIG.

We thank NATO for a grant for international collaboration in research (for A.G. and G.M.)

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