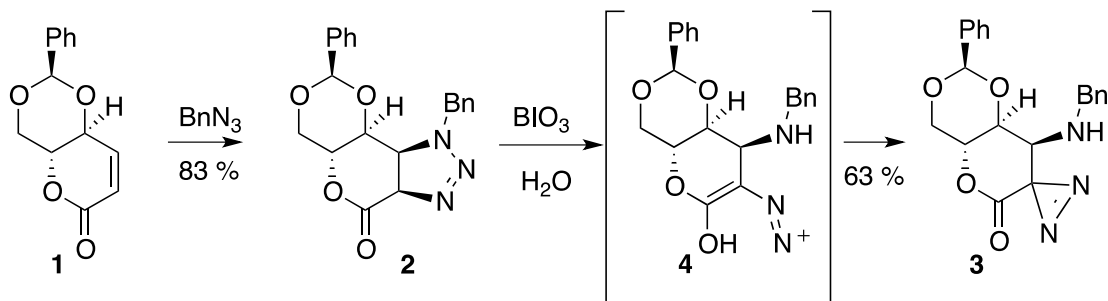


## SYNTHESIS OF (2'*R*,4*A'**R*,8*A'**S*)-8'-(BENZYLAMINO)-2'-PHENYLDIHYDRO-4'*H*-SPIRO[DIAZIRINYL-1,7'-PYRANO[3,2-*D*][1,3]DIOXIN]-6'(4*A'**H*)-ONE

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$\delta$ -Lactone **1** was refluxed before with benzylazide in trimethyl orthoformate giving a single product identified as triazoline **2**. In an attempt to cleave the acetal moiety triazoline **2** was suspended in H<sub>2</sub>O/acetonitrile mixture (8/2) with catalytic amount of BiO<sub>3</sub> and heated under reflux for 2h. Unexpectedly, after neutralization and evaporation the crude material gave diazirine **3**, as a virtually pure product in 63 % yield. Looking at lit<sup>[1]</sup> it was found that diazodixanediones (similar to **4**) isomerizes under photochemical conditions, at 355nm, to give mainly the corresponding diazirines. Also, the diazirines may give back the diazo compounds either under thermal or photochemical conditions. Our mechanistic proposal for the formation of **3** is based in the diazo-diazirine isomerization: intermediate **4** would be generated first and then cyclize to diazirine **3** according to scheme 1. Diazirine **3** was unequivocally identified by spectroscopic means.



Scheme 1

### References:

- [1] Aneta Bogdanova and Vladimir V. Popik *J. Am. Chem. Soc.*, **2003**, 125, 1456–1457.