

Tárgy Organic & Biomolecular Chemistry - Decision on Manuscript ID OB-ART-03-2013-040547



Feladó <obc@rsc.org>

Címzett <borbas.aniko@pharm.unideb.hu>

Dátum 2013-04-16 09:46

16-Apr-2013

Dear Dr Borbás:

Manuscript ID OB-ART-03-2013-040547

Title: Systematic study on free radical hydrothiolation of unsaturated monosaccharide derivatives with exo- and endocyclic double bonds

We have now received the reviewers' reports on your manuscript, which are copied below. Both reviewers feel that the manuscript is potentially suitable for publication in Org. Biomol. Chem., however, referee 2 has raised some fundamental issues that would need to be addressed before publication can be considered. Referee 1 also indicated some points for revision.

Based on the reviewers' comments publication of your manuscript in its present form is not possible. However, providing that the comments of the reviewers are addressed in full, I will be pleased to consider a revised manuscript. The manuscript may be subject to further peer review prior to reaching a final decision on acceptance.

You can submit your revised manuscript through your Author Centre at <http://mc.manuscriptcentral.com/ob>. You will find your manuscript title listed under "Manuscripts with Decisions." Under "Actions," click on "Create a Revision." Alternatively, follow the link below to create your revision:

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Please provide the following as separate files:

- A suitably revised manuscript in native format (.DOC or TeX) with high quality embedded figures

OR

- A suitably revised manuscript in native format (.DOC or TeX) with numbered figures, as separate files in .TIF, or .EPS format, with resolution of 600 dpi or greater and schemes preferably as ChemDraw files. Chemwindow files (filename.cwg, filename.cw2), ISIS/Draw exported as sketch format (filename.skc) and ChemSketch exported in ChemDraw format (filename.cdx) may also be supplied. Providing the original figure and scheme files in their native formats will assist us in ensuring the best quality production of your article.

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- The RSC encourages the submission of supporting information for compounds and spectra in electronic format, and these will be used to enhance the published article. For compounds, please supply mol files (exported from your chemical drawing package) as unique compounds,

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Once again, thank you for submitting your manuscript to Organic & Biomolecular Chemistry and I look forward to receiving your revision.

Yours sincerely,
Elisa

Dr. Elisa Meschini
Publishing Editor - Synthetic Organic Journals
Royal Society of Chemistry
Thomas Graham House
Science Park
Cambridge CB4 0WF, UK
Tel +44 (0)1223 432696
Fax +44 (0)1223 420247
<http://www.rsc.org/publishing>

Reviewer(s)' Comments to Author:

Referee: 1

Comments to the Author

In this manuscript Borbas and co-workers describe an in-depht study on thiol-ene ligations involving sugar derivatives as the ene-partner. This chemistry has not been very exploited in carbohydrate derivatives and the authors show in this paper its considerable potential. The study addresses most of the important issues in this chemistry, and it is noteworthy the reaction of thiol-ene reaction of sugar-endocyclic double bonds, where the the reaction of Neu5Ac-2-ene (17) with a 6-thio galactopyranose derivative is a case in point. In my opinion this manuscript reports on the scope and limitations of a very useful strategy for ligation of unsaturated sugar derivatives to thiols which could grant access to biologically relevant molecules, and deserves to be published in Organic Biomolecular Chemistry.

Some minor details to be addressed prior to publication:

The authors might include their recent publication in Synlett (2013) in the reference section.

Page 2 Line 25: Include (DPAP) after 2,2-dimethoxy-2-pheylacetophenone

Page 3 Line 45-46: give rise to a noticeable increase

In Scheme 4, please include also the yield in the Scheme.

Page 5, Line 18: monosaccharides

Referee: 2

Comments to the Author

The manuscript by Borbas and co-workers reported the study of radical hydrothiolation of unsaturated monosaccharide derivatives with exo- and endocyclic double bonds. The work was extended from the authors' earlier communication reported in OL by the same lab (ref. 16), and this full article was supposed to be a more systematic study as indicated in the title. But some important issues were not addressed. For example, in table 1, the exclusive 1,2-

cis stereochemistry of the reaction is worth some discussions. It is known that the reaction is heavily solvent-dependent. In this lab's earlier work (ref. 16), it was shown that toluene is not the optimal solvent in many cases. The current work didn't answer this fundamentally and practically important question: how solvent affect reaction outcome and what is the guideline for solvent selection? DMF-water system worked very well for some cases where toluene failed, then what happens for the low yield reaction in Table 1 if switch solvent to other systems such as DMF-water? The authors rationalized the different results of different cases as the results of the different stability of the thiyl free radicals, but I could see why 2d is more stable than 2c (the free radical center is not at the benzylic carbon), and why 2e and 2g are significantly more stable than 2f?

In table 2, the authors attributed the lower yield of 5a compared to that of 3a as a result of the lability of the Bn protecting group under UV irradiation. However, the Bn's should be stable under the irradiation conditions with irradiation wavelength of 365 nm (ref. 23-25). It is probably the radicals caused the trouble. A control experiment would clearly show whether 4 is stable under the irradiation condition in the absence of RSH and DPAP. In addition, there are some other minor errors such as:

1. Page 1, abstract, line 8, and page 2, column 1, line 28, in "cleavable photoinitiator", "cleavable" is redundant.
 2. Page 1, column 1, line 32, polymeric?
 3. Page 2, column 1, line 11, mimics?
- and more.

The results are worth publication after the concerns above are fully addressed.