

Pergamon

Corrigendum

Corrigendum to "A sensitive visual test for detection of -OH groups on resin"

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Recently, we published in this journal a paper that described a method to visualize the presence of OH groups on a resin.1 The method was based on the so-called Procion-dye technique, where evanuric chloride (trichlorotriazine, TCT) is used to link a dye to a cellulose fibre. Resin beads were treated with TCT in THF followed by a nucleophilic dye. If an OH group was present on the resin, the beads take the colour of the dye. In that paper, we also reported that the method could be used to monitor the effective loading of a molecule on a Wang-type resin. We carried out some experiments using the same Wang resin purchased from a supplier, that resulted positive with our test and negative after the loading of a carboxylic acid.

As soon as the paper was published, we received several E-mail messages from researchers who had attempted the test on Wang resin and were not able to repeat our experiments. From this correspondence and from several additional experiments done in our laboratories, we found that Wang resins of different quality, obtained from different suppliers, behaved differently in our test.

As the main problem seemed to be the formation of the first intermediate (loading the TCT on the resin) we tried to optimise the conditions to verify if it was possible to obtain reproducible results with different batches of Wang resin.

We report here the correct conditions to yield a positive test on a Wang resin (as on any other resin with a free OH) that allows monitoring the loading of the first molecule on it. The main difference, with respect to the previous protocol, is the use of DMF in the first step instead of THF and the heating of the test tube at 70°C. After this treatment, different kinds of dyes can be attached to the resin. For example, using alizarin R (AliR or Mordant Orange 1, C.I. 14030) the beads appear red, with fuchsin (C.I. 42510) they appear fuchsia and with fluorescein they become fluorescent

The correct protocol is as follows:

- 1. Take some beads of the resin (swollen) and introduce them into a test tube. Wash the beads several times with DMF.
- 2. Add 3 mL of DMF followed by 1 mL of NMM and 5 mg of solid TCT.³
- 3. Heat the test tube at 70°C for 20 min using an oil bath.
- 4. Remove the solution and rinse the beads several times with DMF.
- 5. Add 3 mL of DMF followed by 5 mg of AliR (or 3 mL of a 0.025% solution of fuchsin in NMP or a 0.025% solution of fluorescein in NMP) and 1 mL of NMM.4
- 6. After 5 min, discard the solution and wash the beads with DMF until the solvent is clear. Wash finally with THF or CH₂Cl₂. Observe the colour of the beads directly in the test tube or with a microscope.

This protocol is more time consuming than the other but can be successfully applied to any kind of Wang resin compared with other cases where the previous (more rapid) conditions may be unsuccessful.

References

- 1. Attardi, M. E.; Falchi, A.; Taddei, M. Tetrahedron Lett. 2000, 41, 7395-7399.
- 2. Allen, R. L. M. Colour Chemistry; Appleton-Century-Crofts: New York, 1971.
- 3. Following this procedure, the chlorobenzyl derivative of the Wang resin was not formed as demonstrated by microanalysis of the beads where nitrogen was present. For the transformation of alcohols in chlorides using TCT, see: Sandler, S. R. J. Org. Chem. 1970, 53, 3967.
- 4. The solutions of fuchsin or fluorescein must be prepared just before their use.

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