

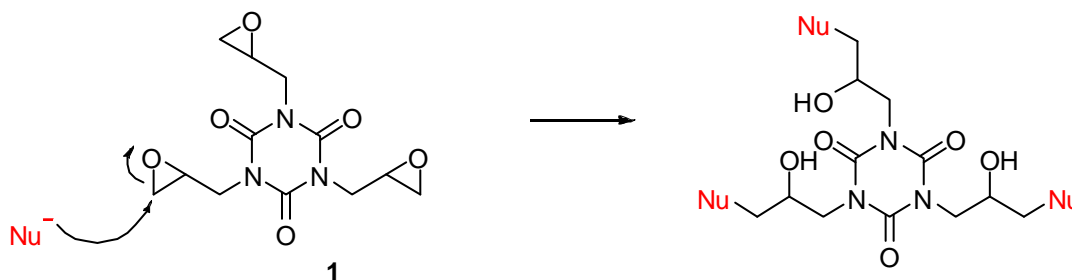
Synthesis of triols with C₃ symmetry by ring opening of tris(oxiran-2-ylmethyl)-1,3,5-triazinane-2,4,6-trione with nucleophiles

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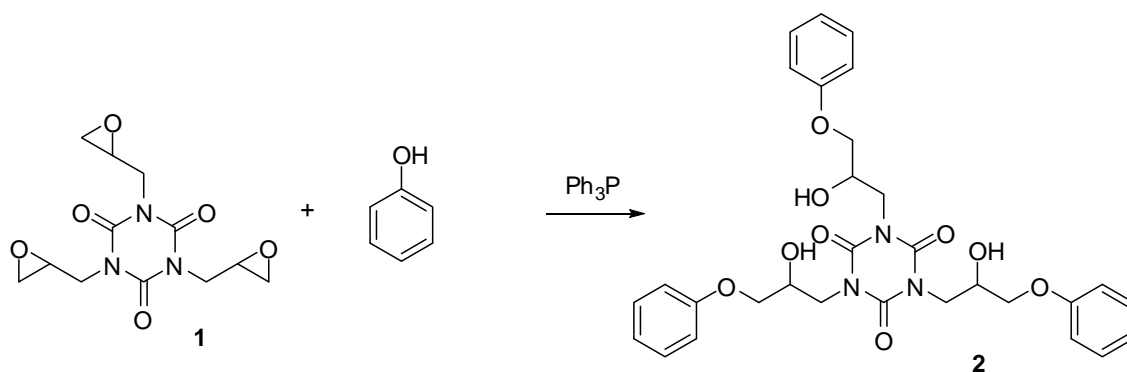
Abstract: The reaction of tris(oxiran-2-ylmethyl)-1,3,5-triazinane-2,4,6-trione with phenol or N-methylaniline promoted by microwave heating, under solventless conditions, led to the opening of the oxirane ring through the less hindered carbon, leading to tripodal triols with C₃ symmetry in good yields.

Tris(oxiran-2-ylmethyl)-1,3,5-triazinane-2,4,6-trione (**1**) usually known as 1,3,5-triglycidyl isocyanurate (TGIC) is a common ingredient in polymer formulations as cross linking agent.¹ It also constitutes an interesting nucleus for the preparation of compounds with C₃ symmetry, since the oxirane ring is very suitable for opening with nucleophiles leading to substituted triols (Scheme 1).



Scheme 1.

In this communication, TGIC was treated with aromatic nucleophiles. As model compound phenol was used, this together with TGIC (3:1 ratio) was heated at 130 °C, under argon atmosphere, for ten minutes. Then, a catalytic amount of triphenylphosphine was added and the mixture heated for 20 minutes. Once purified, it yielded 84% of 1,3,5-tris(2-hydroxy-3-phenoxypropyl)-1,3,5-triazinane-2,4,6-trione (**2**).

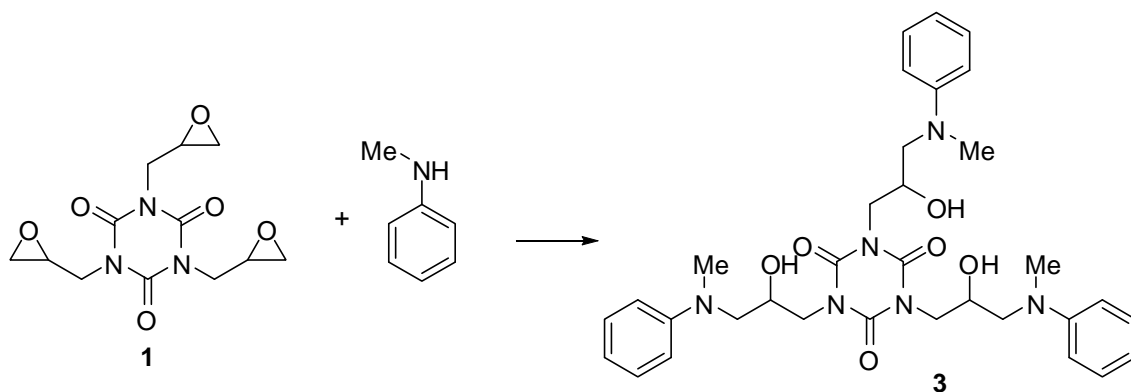


Scheme 2.

The compound was characterized by its spectroscopic data. The mass spectrum displayed the molecular ion at m/z 579. The IR spectrum indicated the presence of a hydroxyl group at 3273 cm^{-1} . The ^1H NMR spectrum showed signals characteristic of aromatic hydrogens, and four double-doublets (δ 4.18, 4.14, 3.77 and 3.63 ppm) for each of the diastereotopic hydrogens $\text{CH}_2\text{CH}(\text{OH})\text{CH}_2$.

The feasibility of its synthesis by microwave irradiation was studied, because as it is well known,² in many cases microwaves reduce the formation of secondary products and speed up the reactions. In this case TGIC, phenol and triphenylphosphine were mixed in the same proportions as in the conventional method, and irradiated in a monomode oven (CEM, Discover) at $130\text{ }^\circ\text{C}$ and 300 W for 10 minutes. The crude, after purification, rendered **2** in 80% yield.

The opening of the oxirane ring in TGIC was also studied with a nitrogenated nucleophile: N-methylaniline. Using conventional conditions, a 3:1 mixture of N-methylaniline and TGIC, was heated at 140°C for 30 minutes under argon atmosphere, after purification **3** was isolated in 52% yield (Scheme 3). Its structure was identified from its spectroscopic data. Mass spectrometry displayed a peak at m/z 619 consistent with the molecular ion plus one unit. The ^1H NMR spectrum shows the aromatic hydrogens, a singlet for NCH_3 group (δ 2.98 ppm), a multiplet (δ 4.24 ppm) corresponding to CHOH and four double doublets of diastereotopic hydrogens on alpha to this carbon (δ 4.12, 3.96, 3.43 and 3.34 ppm).



Scheme 3.

The process was improved by irradiation with microwaves (300W, 110°C, 10 minutes), rendering, after purification, 90% yield.

In resume, the epoxide ring opening of tris(oxiran-2-ylmethyl)-1,3,5-triazinane-2,4,6-trione (**1**) with phenols or anilines is a suitable method to obtain tripodal triols. This was carried out by conventional heating or microwave irradiation, showing that the last one allowed shorter reaction times and better yields.

Acknowledgements

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References

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