

Correction to “Comprehensive Synthesis of Monohydroxy–Cucurbit[*n*]urils (*n* = 5, 6, 7, 8): High Purity and High Conversions”

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J. Am. Chem. Soc. **2015**, *137*, 10238–10245. DOI: [10.1021/jacs.5b04553](https://doi.org/10.1021/jacs.5b04553)

S Supporting Information

During our follow-up work on the conjugation of monohydroxylated cucurbiturils, we found that the reported yields of monohydroxylation of CB[*n*] (*n* = 5–8), 90–95%, were incorrect. This was due to the misleading composition of the crude product, with an approximate 0.3/0.3/0.3 ratio of CB[*n*], CB[*n*–(OH)₁], and CB[*n*–(OH)₂], giving NMR signal integrals and peak positions that match the expected values for pure CB[*n*–(OH)₁]. After purification of the product via silica column chromatography using water/acetic acid/formic acid as eluent, the corrected yields are 5–37% for CB[*n*–(OH)₁], with *n* = 5–8. These results were confirmed by repeated experiments starting with 100 mg, and using up to 3 g, of CB[*n*].

The Supporting Information has been revised to include MS spectra of pure CB[*n*–(OH)₁ and details of the column chromatography purification procedure for all CB[*n*–(OH)₁.

Page 10238. The title should be revised to Comprehensive Synthesis of Monohydroxy–Cucurbit[*n*]urils (*n* = 5, 6, 7, 8).

Page 10238. In the Abstract, the 95–100% conversion reported for CB[*n*–(OH)₁ should be corrected to 20–40%, depending on the CB[*n*] considered. Scale-up experiments were performed using up to 3 g of CB[*n*] with quartz reactors of 50 or 300 mL.

Page 10239. In the Synthesis section, the conversions given in the text and in Table 1 are incorrect due to the misleading ¹H NMR spectra, and should actually be 20–40%. The values in Table 1 are thus apparent conversions determined by ¹H NMR. Isolated yields have been determined and are given in the revised Supporting Information (5–37% for the CB[*n*–(OH)₁ series).

Page 10242. In Table 3, likewise, the conversion of CB[8] toward the formation of CB[8]–(OH)₁ is an apparent conversion determined by ¹H NMR, where CB[8] is transformed to CB[8]–(OH)₁ and CB[8]–(OH)₂. Thus, the title of the table should be revised to “Apparent Conversion of CB[8] toward the Formation of CB[8]–(OH)₁”.

■ ASSOCIATED CONTENT

S Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: [10.1021/jacs.6b00188](https://doi.org/10.1021/jacs.6b00188).

Procedure for the preparation of CB[*n*–(OH)₁, ¹³C NMR spectra, details of high resolution MS analyses, decay products of CB[8], details for the calculations of BDEs, and structures of each CB[*n*]· radical (revised) (PDF)

Published: February 3, 2016