The absolute configuration of (+)-(*E*)-4-phenylbut-3-ene-2-ol

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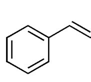
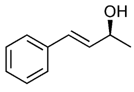
A derivative of (+)-(*E*)-4-phenylbut-3-ene-2-ol is shown by X-ray crystallography to be of (*R*) configuration, confirming the assumption in the literature that the absolute configuration of (+)-(*E*)-4-phenylbut-3-ene-2-ol is (*R*).

(*E*)-4-Phenylbut-3-ene-2-ol **1** is widely used to exemplify methods in enantioselective synthesis, for example as a target for enantioselective reduction of carbonyl compounds,[1] or a substrate for kinetic resolution[2] or dynamic kinetic resolution[3] methods. Indeed, a quick *Chemical Abstracts* search reveals over 100 papers that refer to isomerically pure **1** of either configuration.


(R)-**1**

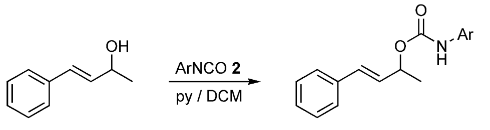

(S)-**1**

When starting work with **1** we decided to check the data used to determine the absolute configuration of the alcohol. For example, a recent communication from Tiecco *et al.* states that the absolute configurations of **1** "were assigned by comparison of the sign of the optical rotations with those reported in the literature"[4] and cite a paper by Kamal *et al.* from 2003,[5] which, in common with many others, cites Burgess *et al.* from 1991.[2] The Burgess group compared their optical rotations with another widely cited paper, published by Terashima *et al.* in 1980.[1]

Following this literature trail to its end, we noted that Terashima *et al.* referred back to 'Absolute Configurations of 6000 Selected Compounds with One Asymmetric Carbon Atom',[6] but were dismayed that the reference for the configuration of **1** was a simple note "Mme Gouket and H. Felkin". Hence, the >100 papers mentioned above have been working on the assumption that Mme Gouket and H. Felkin were correct, with no knowledge of the experimental methods they used.

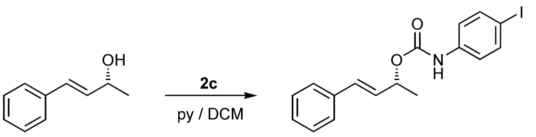
Single crystals of the alcohol (+)-**1** suitable for X-ray diffraction were grown from an ethyl acetate solution. The X-ray crystal structure of these was solved using copper radiation,† but the value of the Flack parameter could not be determined reliably enough to be sure of the absolute configuration of the alcohol, presumably because (+)-**1** only contains one oxygen atom.

Reaction[7] of (+)-**1** with isocyanates **2** yielded the expected (+)-carbamates **3** as crystalline solids suitable for X-ray crystallography.[8] Of these, carbamate **3c** was most suitable for determination of the configuration. The value of the refined Flack parameter was 0.01(4) for (*R*)-(+)-carbamate **3c**.



(+)-**1**
(+)-**3** **a**; Ar = 4-bromophenyl
b; Ar = 2,4,6-tribromophenyl
c; Ar = 4-iodophenyl

Therefore, assuming that the absolute configuration of **1** is unchanged during reaction with **2c**, we can conclude that Mme Gouket and H. Felkin were correct to state that (+)-(*E*)-4-phenylbut-3-ene-2-ol is of (*R*) configuration.



(+)-**1**
(+)-**3c**

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Notes and references

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6 J. Jacques, C. Gros and S. Bourcier, *Absolute Configurations of 6000 Selected Compounds with One Asymmetric Carbon Atom*, Thieme, Stuttgart, 1977, vol. 4.
7 D. B. Smith, Z. Wang and S. L. Schreiber, *Tetrahedron*, 1990, **46**, 4793.
8 Single crystals of the carbamate $C_{17}H_{16}NO_2I$ **3c** were grown from a solution in acetonitrile, mounted in an inert oil and transferred to the cold gas stream of the diffractometer. Crystal data. Compound (*JM2*) $C_{17}H_{16}NO_2I$, $M = 393.21$, monoclinic, $\beta = 102.311(2)°$, $a = 8.9131(9)$, $b = 5.0982(5)$, $c = 18.2642(17)$ Å, $U = 810.85(14)$ Å^3, $T = 180$ K, space group $P2_1$ (no. 4), μ(Mo–Kα) = 1.978 mm^{-1}, $Z = 2$, $\lambda = 0.71073$ Å, 5524 reflections measured, 3697 unique [$R_{int} = 0.0345$] which were used in all calculations. The final $wR(F_2)$ was 0.1258. Refined Flack parameter 0.01(4) on 1492 Friedel pairs. CCDC reference number 601966. For crystallographic data in CIF or other electronic format see DOI: 10.1039/b603934k.