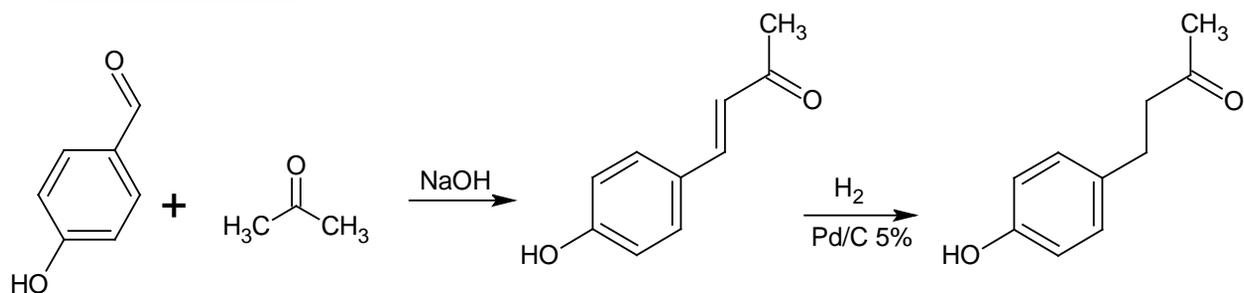




Introduction:

Rheosmin (Raspberry Ketone) is a natural product responsible in part for the smell of raspberries. It is used in many cosmetic and food products because of its fruity odour and flavour.

Reaction Scheme:



Reagents:

- | | |
|--------------------------|--------------|
| i) 4-Hydroxybenzaldehyde | Acros 99% |
| ii) Propanone | Fisons 99.5% |
| iii) Sodium Hydroxide | Fisher 98% |
| i) Palladium / Carbon 5% | J&M 98% |
| ii) Hydrogen | BOC 99.992% |
| iii) Ethyl Acetate | Fisons HPLC |
| iv) Sodium Acetate | OLI 98% |



Experimental:

10g. 4-Hydroxybenzaldehyde was dissolved in 40ml of CaO dried Propanone. Dissolution was accomplished with stirring. An orange solution was obtained. (Fig. 1)



Fig. 1

Experimental:

Slowly, a 10% solution of Sodium Hydroxide in water (4g in 40ml) was dripped in. The solution became darker and was left to stand overnight, the following morning a large mass of stunning red/orange crystals had formed. (Fig. 2)



Fig. 2

These were slurried into 200ml, ice cold 10% HCl forming a dark oil, which crystallised spontaneously. (Fig. 3)



Fig. 3

The above crystals were filtered, and recrystallised from boiling water, forming stunning yellow/white needles. (Fig. 4)

Yield- 5g. Recrystallised, melting point was 124 C sharp.



Fig. 4

A Hydrogenation set up compiled of a flask flushed with hydrogen attached to an upturned gas jar filled with hydrogen. Into which was added the above product dissolved in 20ml. Ethyl Acetate, 5.125g Sodium Acetate and 0.5g 5% Pd/C. (Fig. 5)



Fig. 5

The slurry was allowed to stir and to absorb Hydrogen, a total of 800ml Hydrogen was absorbed over the course of 4 hours, after which an abrupt cessation of absorption indicated a completed reaction. This extra-ordinarily large amount of Hydrogen implied a leak in the glassware- this was to be expected. (Fig.6)



Fig. 6

Once Hydrogen absorption ceased, the contents of the reaction vessel were vacuum filtered under a flow of Nitrogen, the catalyst recovered and the filtrate washed with 2x20ml Water in order to remove the Sodium Acetate. The Ethyl Acetate was rotavaped to yield a strongly smelling oil which crystallised spontaneously. (Fig 7,8)

Yield 4.12g Melting point was not satisfactory

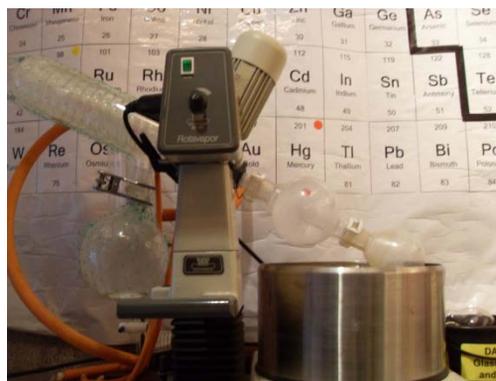


Fig. 7

This was recrystallised from 10ml boiling water, yielding 3.43g of beautiful, faintly smelling yellow/white crystals with a melting point of 82 C sharp. A small quantity was ampouled for further analysis (Fig. 9)



Fig. 8

References

Our sincere thanks to Klute for the idea and reduction technique.



Fig. 9