Oriental Journal of Chemistry Vol. **26**(1), 189-193 (2010)

Synthesis of 2-pentyloxy allyl ester

C. RAVICHANDRAN

Department of Chemistry, Easwari Engineering College (SRM Group),
Ramapuram, Chennai - 600 089 (India).

(Received: November 05, 2009; Accepted: December 19, 2009)

ABSTRACT

Chemical synthesis is purposeful execution of chemical reactions in order to obtain a product, or several products. It begins by selection of compounds that are known as reagents or reactants. Organic chemistry is a specific discipline within chemistry, which involves the scientific study of the structure, properties, composition, reactions and preparation by synthesis of chemical compounds. In view of the demand and utilization, 2-pentyloxy allyl ester (Allyl Amyl Glycolate - AAG) has been identified for the present investigation and synthesis. During the synthesis, the effect of various components namely reactants, temperature, catalyst etc were studied. Tentatively, the optimum operating parameters have been arrived at.

Key words: Iso amyl alcohol, Intermediate acid and fragrance.

INTRODUCTION

Perfumes are thousands of years old - the word "perfume" comes from the latin word 'perfume' meaning "through smoke"[1]. Fragrance chemistry, together with the closely related area of flavour chemistry, is one of the few domains, if not the only one, in which chemists can immediately experience structure – activity relationships. Today there are over 20,000 perfume fragrances in the market. The perfume industry has undergone several changes in technique, material and style, which have led to the creation of modern perfume fragrance industry, one that still incorporates creativity, mystique and romance along with marketing to appeal to the masses. The term fragrance or aroma is used primarily by the food and cosmetic industry to describe a pleasant odour[2]. Creating perfumes from organic substances, such as plant and animal scents[3], is an expensive process that results in very price perfume.

Synthetic aromas in perfumes are not normally found in the environment, but are common in many of today's perfumes. Synthetics come from combinations of some of the chemicals found in petroleum and pine. One such example is the pale yellow liquid ionone, with its woody, dry, fruity, raspberry, and violet aromas[4&5]. Since the allyl amyl glycolate has a characteristic fragrance and it can be used to reinforce modern green and oriental fragrance with it's tenacity, it is markedly useful as a scenting compound of various fragrances & cosmetics and a powerful fruity and pineapple note with green galbanum nuances.

EXPERIMENTAL

To a 2-liter three-necked round bottom flask fitted with a stirrer and condenser was charged 1 liter of iso amyl alcohol. It was heated to around 60 degree Celsius and sodium methoxide was added slowly over a period of 1 hour and at a later

stage it may be necessary to increase the temperature for the dissolution of sodium methoxide. After the addition of sodium methoxide, the by-product methanol was distilled to collect around 300 ml of the distillate. By now the temperature would have reached above 130 degree Celsius. 200 ml of fresh iso amyl alcohol was added and thereafter addition of sodium mono chloro acetate started[6].

The addition has to be made over a period of 45 minutes and at the same time reflux of iso amyl alcohol should be maintained. After addition, it was further refluxed for about 2 hours. Finally it was cooled and transferred to a metal vessel. Upon cooling, 1 liter of ice water was added and the mixture was transferred to a plastic container. For dissolving the intermediate salt, additional water may be added if required. The intermediate salt was acidified using concentrated HCl and finally washed with salt water twice. Iso amyl alcohol was recovered by distillation and the intermediate crude acid was distilled to obtain around 300 grams of intermediate acid containing some unwanted ester.

The intermediate acid was taken along with 300 ml of benzene, allyl alcohol and catalyst. Esterification water was removed azeotropically. The reaction mixture after removing the expected amount of water was cooled and washed with sodium bicarbonate followed by water to neutral pH. Benzene was distilled and then the product was fractionated carefully using a long packed column to obtain pure Allyl Amyl Glycolate. The yield of pure AAG was 52%. The purity of the compound was around 99% by Gas chromatography.

RESULTS AND DISCUSSION

Aroma compound also known as odorant, aroma, fragrance, flavour, is a chemical compound that has a smell or odour. Fragrance chemistry, together with the closely related area of flavour chemistry, is one of the few domains, if not the only one, in which chemists can immediately experience structure – activity relationship[7].

Table 3.1: Effect of iso amyl alcohol with constant temperature and sodium methoxide

S. No	Weight of iso a myl alcohol(liter)	Weight of sodium methoxide(gm)	Temperature (degree celsius)	Weight of intermediate yield(%)
1	0.250	110	120-125	5.6
2	0.500	110	120-125	12.8
3	0.750	110	120-125	28.7
4	1.000	110	120-125	92.7
5	1.250	110	120-125	90.0
6	1.500	110	120-125	81.0

Table 3.2: Effect of sodium methoxide with constant temperature and iso amyl alcohol

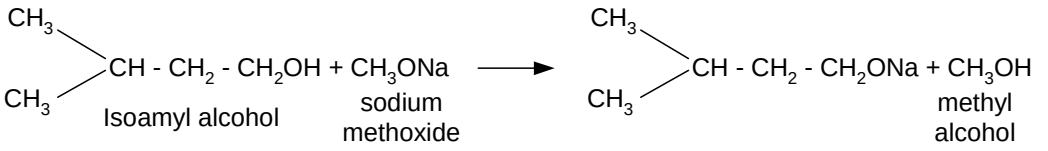
S. No	Weight of iso a myl alcohol(liter)	Weight of sodium methoxide(gm)	Temperature (degree celsius)	Weight of intermediate yield(%)
1	1.00	70.0	120-125	24.0
2	1.00	80.0	120-125	28.3
3	1.00	90.0	120-125	36.1
4	1.00	100.0	120-125	52.0
5	1.00	110.0	120-125	82.0
6	1.00	120.0	120-125	70.0

The fusion of science and technology is what fuels creative and revolutionary thinking at technology flavors and fragrances. The end result is the high quality flavors and fragrances for a wide range of innovative products[8&9].

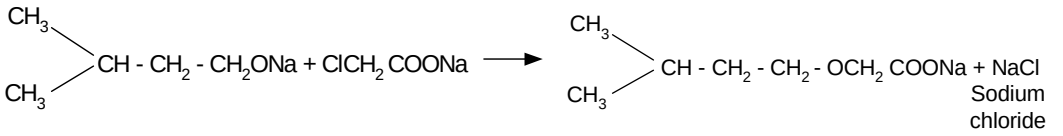
The allyl amyl glycolate were synthesized according to the procedures from the literature. Based on our practical experience and experimental results, we made some changes in the procedures, which marginally increased the rate of the reaction. The concentration effect of iso amyl alcohol was studied with constant quantity of sodium methoxide at constant temperature (Table 3.1). It shows that with 1liter of iso amyl alcohol and constant parameters, 92.7 percentage of intermediate yield could be achieved.

Table 3.2 shows the effect of sodium methoxide with constant quantity of iso amyl alcohol at constant temperature. The effect of sodium methoxide reveals the effect of carbanion in accord with views on the cleavage of a covalent bond. 110g of sodium methoxide produced 82% of intermediate yield[10].

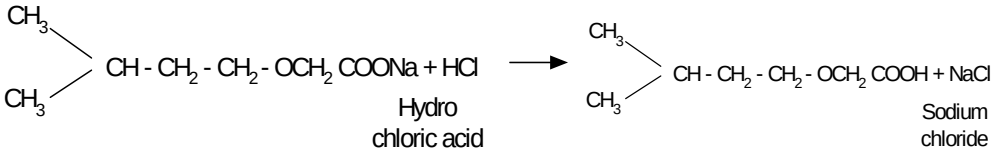
Reaction mechanism



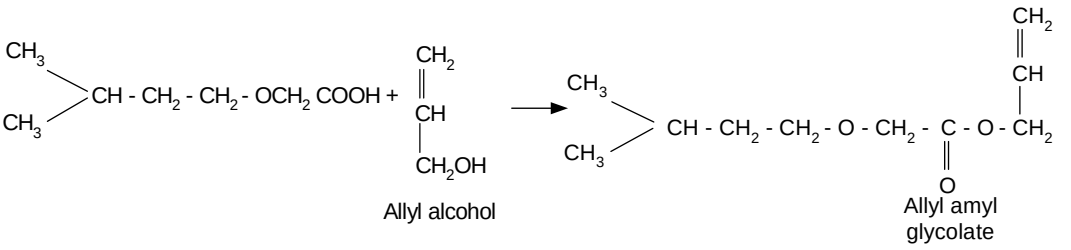
Step 1



Step 2



Step 3



Step 4

The effect of temperature (Table 3.3) shows that at 120-125 degree Celsius, maximum intermediate yield of 99.3% was produced.

Table 3.4 shows the effect of allyl alcohol, which is used as a medium with the intermediate acid. The concentration of 80% allyl alcohol will produce the maximum yield of 48.5%. If the concentration level is increased, the yield percentage is decreased. This may be due to the unsaturation effect of the allyl alcohol. The optimum concentration will be fixed as 80% [11].

The effect of intermediate acid with constant allyl alcohol and temperature studies, gives an idea about the concentration change in the system and the maximum percentage of yield was achieved at 60% of intermediate strength (Table – 3.5). If the concentration level of the acid is increased, the product yield percentage decreased. Finally, it was found that the optimum operating parameters were Iso amyl alcohol: 1 liter, Sodium methoxide: 110g, Temperature: 120-125 degree celsius and Allyl alcohol: 80%. The yield of AAG was 52%. The physical and chemical properties of

Table 3.3: Effect of temperature with constant iso amyl alcohol and sodium methoxide

S. No	Weight of iso a myl alcohol(liter)	Weight of sodium methoxide(gm)	Temperature (degree celsius)	Weight of intermediate yield(%)
1	1.00	110	100-105	24.3
2	1.00	110	105-110	32.0
3	1.00	110	110-115	39.0
4	1.00	110	115-120	67.6
5	1.00	110	120-125	99.3
6	1.00	110	125-130	91.0

Table 3.4: Effect of allyl alcohol with constant temperature and intermediate acid

S. No	Weight of iso a myl alcohol(liter)	Weight of allyl alcohol (liter)	Temperature (degree celsius)	Weight of intermediate yield(%)
1	60	50	120-125	12.0
2	60	60	120-125	14.2
3	60	70	120-125	19.5
4	60	80	120-125	48.5
5	60	90	120-125	43.0
6	60	100	120-125	41.0

Table 3.5: Effect of intermediate acid with constant temperature and allyl alcohol

S. No	Weight of iso a myl alcohol(liter)	Weight of allyl alcohol (liter)	Temperature (degree celsius)	Weight of intermediate yield(%)
1	30	80	120-125	11.4
2	40	80	120-125	14.5
3	50	80	120-125	20.6
4	60	80	120-125	49.5
5	70	80	120-125	43.0
6	80	80	120-125	41.0

allyl amyl glycolate was comparatively studied and confirmed with the literature.

The effect of intermediate acid, benzene, ally alcohol and catalyst and also the esterification process of the system will be studied in our further course of studies to increase the quantity of final product.

CONCLUSION

The preparative method for the synthesis of allyl amyl glycolate from iso amyl alcohol described here is a simple and environment-friendly technique, which can be commonly adopted for synthesis of various compounds. The present study suggests the optimum operating parameters.

ACKNOWLEDGEMENTS

The author wishes to express his sincere thanks to the Principal, Director and the SRM Management for permission to publish this paper.

REFERENCES

1. Richard F and Sally J.Daley, Organic chemistry textbook, 2006.
2. Edward, Michel, " Perfume Legends: French Feminine Fragrances". Crescent House Publishing (1997).
3. Aroonrat Meekijjaroenroj, Jean-Marie Bessiere, Marie-Chrlotte Anstett, *Flavour and Fragrance Journal,* **22**(4): 300-310 (2007).
4. Moran, " Fabulous Fragrances II: A Guide to prestige Perfumes for Women and Men" Crescent House Publishing, (2000).
5. Turin, Luna, " The secret of scent", Faber and Faber (2006).
6. Marc Leudon, Organic chemistry, 4th edition, Oxford university press, New York.
7. Kun – Hosong and Kwang – Rae Lee, *Korean J.Chem.Engg*., **22**(5): 735 (2005).
8. Burr and Chandler, The Emperor of scent: The story of Perfume, Obession and the last mystery of the senses. Random house, New york (2003).
9. Na Shu and Hong Shen, Research Articles, *Flavours and Fragrance journal,* **24**(1): 1- 47 (2009).
10. Turova, N.Ya. Metal oxoalkoxides, Synthesis, properties and structures (Review). Russian Chemical reviews, **73**(11): 1041-64 (2004).
11. Norman and coxon, Principles of organic synthesis. 3rd edition, Chaparan and Hall (ELBS).
12. Calkin, Robert R and Jellinek, J Stephen, "Perfumery: Practice and Principles". John Wiley and Sons, Inc., (1994).
13. E.W. Dean and D.D. Stark, *Ind. Engg. Chem.*, **12**(5): 486-490 (1920).
14. V.K.Ahluwalia and Rence Aggarwal, Organic synthesis, special, techniques, Narosa publishing house, New Delhi.
15. Organic syntheses, a publication of reliable methods for the preparation of organic compounds.