

## **Economical and Simple Preparation of Oleyl Bromide from Commercially Available Olive Oil**

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### **Authors' contributions**

*This work was carried out in collaboration between all authors. Authors JMM, IAC and MDP designed the study. Authors FJC, CMU and REF carried out the synthesis, purification and characterization of all compounds. Authors MDP, IAC and FJC designed the synthetic route, managed the literature searches and wrote the manuscript. All authors read and approved the final manuscript.*

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### **ABSTRACT**

The objective of this work was to get access to a cheap and easy to scale source of oleyl bromide. Starting from commercial olive oil, and using economical reagents and conditions, the conversion was achieved in 5 steps (saponification, distillation, esterification, reduction and bromination), with a good overall yield (30%), providing a good quality final product, which showed a purity of at least 95%, by NMR.

**Keywords:** Bromination; economical; feedstock; oleic acid; olive oil.

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## ABBREVIATIONS

MHz : Megahertz.  
EtOAc : Ethyl Acetate.  
TLC : Thin Layer Chromatography.  
UFA : Unsaturated Fatty Acids.  
PUFA : Polyunsaturated Fatty Acids.

## 1. INTRODUCTION

Fatty acids as organic feedstock, either as alternative internal combustion engine fuel, also known as biodiesel, or as a source of carbon chains used in the manufacture of miscellaneous industrial materials, can be obtained from renewable natural sources, such as vegetable oils, animal or microbial fats and oils, etc. The actual functional starting materials are ordinarily obtained by the transesterification of a naturally occurring fatty acid derivative, such as a fat, wax or oil, with a low molecular weight alcohol, in order to replace all natural glycerol esters, and also to obtain a more volatile and easy to handle product, which can reach the final consumer in a more reliable and consistent form [1-4]. Unsaturated fatty acid derivatives though, have even more interesting potential uses than their mere incineration, as they are also very useful starting materials for the manufacturing of industrial foods, lubricants, polymers, and many other high added value commodities.

Most vegetable oils are obtained by direct pressing and/or solvent extraction from either seeds or fruits of oilseed crops [5]. Many oils used in deep frying are chosen based on their thermal stability and high smoke point, that is the temperature at which, when an oil is heated, it starts producing visible smoke due to volatile substances present, such as free, shorter, fatty acids, and so degrade in air to give a visible soot. The smoke point signals a practical limit to the temperature at which a given fat or oil can be used for cooking. The smoke is also highly combustible, and a main source of accidental fires either in the kitchen, or the industry [6].

In the case of olive oil, it is obtained by pressing or extracting the fruit of the olive tree, and it is characterized by a high level of oleic acid, ranging 55-83%, with ranges of 8-20% for palmitic acid, and 4-21% for linoleic acid [7]. There are several previous reports describing the treatment, separation and obtainment of oleic acid from olive oil and its mill wastewaters, [8-10] which are focused to either recover the edible oil from waste, or to prepare soap from such residues. There is a classical known method for

the separation of lineal carbon chain compounds, from non-lineal ones using an urea extraction crystallization [11,12].

For another project, related with the synthesis of functionalized organic materials, we needed a cheap and abundant source of carbon-chain compounds that could easily be elaborated into molecular spacers and, using simple known methods, be turned into more complex molecules [13], in multigram scale, with a reasonable chemical purity (technical or laboratory grade), for an organic synthetic laboratory use. We needed a long, straight carbon chain with a good leaving group at one end, and also containing a potential second organic function for further elaboration. The simplest idea was to start from an unsaturated fatty acid, convert its carboxylic acid into a good leaving group, and later on to use the unsaturation as a pivot for a functionalization, using for instance a chain metathesis reaction, or an oxidative double bond fragmentation, etc. We chose oleic acid as starting material, but soon were surprised to find out that despite being available from commercial sources, it comes at a rather steep price, so we turn our attention to natural olive oil, purchased from the nearest local market, as a cheap, sustainable, scalable, easy to obtain, and widely available source of oleates, which were then elaborated into useful organic derivatives.

## 2. MATERIALS, METHODS AND EXPERIMENTAL DETAILS

### 2.1 General

The olive oil was purchased from a local supermarket, labeled as "*Aceite de Oliva Extra Virgen*" (Extra Virgin Olive Oil), and the choice was made under the only base of lowest price of all brands available. The <sup>1</sup>H-NMR (Fig. 1) shows the composition of our oil, using DMSO as internal standard. The signal assignment was made according to Knothe et al. [14].

All reagents and solvents used were the cheapest technical grade available. A summary of all reactions used, is shown below (Fig. 2).

FT-IR spectra were recorded on a Thermo Scientific Nicolet iS10 spectrophotometer. <sup>1</sup>H spectra were recorded at 400 MHz, and <sup>13</sup>C spectra at 100 MHz, on a Bruker AVANCE-III HD 400 instrument, with CDCl<sub>3</sub> as solvent and TMS as the internal standard, unless otherwise stated, and measured in ppm (δ) relative to TMS. All

technical-grade organic solvents, preparative “flash” Silica gel 60, and thin layer chromatography (TLC) plates type Silica gel 60 F254 on aluminum sheets, were purchased from local suppliers. Analytical TLC plates were eluted with mixtures of hexane/EtOAc (2:1), and the compounds visualized with an UV lamp, iodine vapors, and/or sprayed with dilute sulfomolybdic reagent (2.5 g of ammonium molybdate, dissolved in 20 ml of water is diluted with 28 ml of sulfuric acid in 50 ml of water, and then diluted to 1000 ml with water, to spray) and heated.

## 2.2 Saponification [15,16]

To a 2 L beaker containing 100 g of commercial olive oil and 400 mL of water, 60 g (1.07 mol) of

KOH were added in several portions, and mechanically stirred at about 70°C for 2 hours. The resulting mixture was allowed to cool to room temperature, and 300 mL of 30% HCl were slowly added. At this point a very thick, whitish emulsion of oleic acid was produced. To that emulsion, 300 mL of benzene were added, and heated to 70°C with vigorous stirring until two phases were clearly separated. The setup was allowed to cool, and transferred to a separatory funnel, the aqueous phase was discarded, and the organic phase was repeatedly washed with water, until neutral to litmus, then with saturated sodium chloride solution, and dried over anhydrous sodium sulfate for 2 hours.

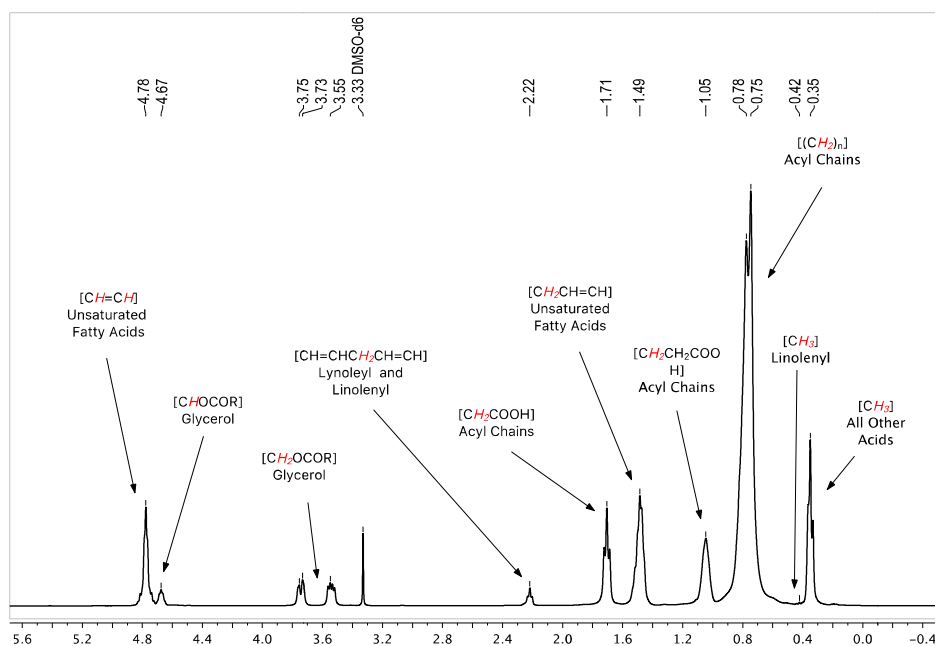


Fig. 1. <sup>1</sup>H-NMR spectrum of starting olive oil (in DMSO-d<sub>6</sub>)

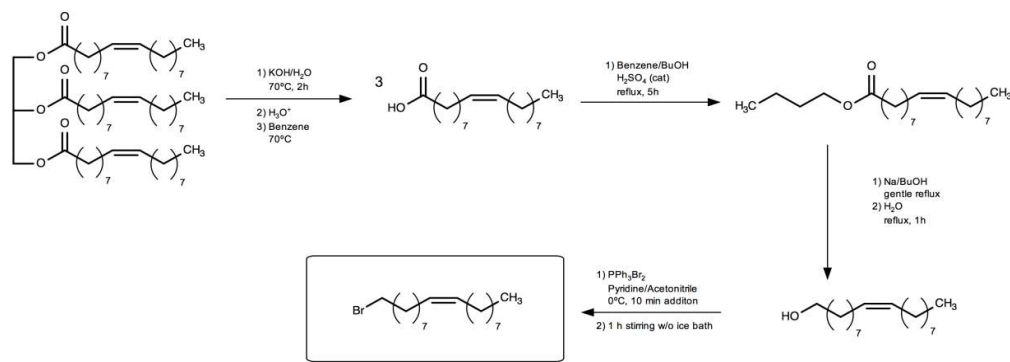


Fig. 2. Chemical transformations

A small portion (2-3 mL) of the organic phase was evaporated under vacuum and dried overnight in a desiccator over phosphorus pentoxide at high vacuum, to obtain a sample of crude oleic acid for characterization purposes. The remaining organic phase was used directly in the next step without any further treatment.

In order to improve the major component purity, the crude hydrolysis product can be distilled, recovering first the benzene to be re-used in the next step, and then reducing the pressure to 0.01 mm Hg, to obtain three fractions distilling at about 155°, 176° and 187° C (bulb temperatures), respectively. The ratio of saturated, monounsaturated and polyunsaturated acids were obtained using <sup>1</sup>H-NMR spectroscopy, by integration of characteristic signals [14].

The forerun fraction (11 g) was a whitish, waxy semisolid at room temperature, and contained the largest ratio of saturated acids. The heart fraction (55 g) was a white, opaque oil at room temperature, containing the highest concentration of Oleic Acid. A tail fraction (18 g), was obtained at 187°C as a yellowish, transparent oil, containing mainly unsaturated and polyunsaturated acids, together with some impurities. The dunder was a gummy, dark brown soft to the touch material at room temperature (6 g). Estimated composition of all fractions is shown in Table 1. Although the different fractions showed diverse compositions, any of them could be used in the next synthetic steps due to their large contents of oleic acid.

All combined fractions weighted 84 g (0.30 mol), i.e.: 88% overall yield, or either 55 g (0.19 mol), 58% yield of pure oleic acid (from the heart fraction).

*Heart fraction:*

<sup>1</sup>H-NMR δ 11.36 (bs, 1H), 5.54 – 5.16 (m, 2H), 2.33 (t, J = 7.5 Hz, 2H), 2.03 (dq, J = 12.3, 6.5 Hz, 4H), 1.63 (p, J = 7.2 Hz, 2H), 1.37 – 1.22 (m, 20H), 0.88 (t, J = 6.7 Hz, 3H).

<sup>13</sup>C-NMR δ 180.64, 130.07, 129.78, 34.24, 32.06, 29.91, 29.82, 29.68, 29.48, 29.47, 29.30, 29.21, 29.19, 27.35, 27.28, 24.79, 22.82, 14.19.

FT-IR (KBr, cm<sup>-1</sup>): ν<sub>O-H</sub> 3083, ν<sub>C-H</sub> 2921, ν<sub>C=O</sub> 1709, δ<sub>C-O</sub> 1285, 1090.

### 2.3 Esterification [17]

The heart fraction obtained in the previous step (50 g, 0.18 mol) was transferred to a 1 L round-bottom flask, provided with a large magnetic bar, and fitted with a Dean-Stark trap and condenser. Benzene (150 mL), butanol (50 mL) and concentrated sulfuric acid (5 mL) were added to the flask and heated to reflux for 5 hours, after which TLC analysis revealed the disappearance of the starting material. The resulting yellowish solution was washed with saturated sodium carbonate, repeatedly washed with water until neutral to litmus paper, and then stirred over anhydrous sodium sulfate for 2 hours, filtered and evaporated *in vacuo* to give a yellowish oily residue, 53 g (0.16 mol), 88% yield.

<sup>1</sup>H-NMR δ 5.26 (s, 2H), 3.99 (t, J = 6.7 Hz, 2H), 2.20 (t, J = 7.5 Hz, 2H), 1.93 (d, J = 6.3 Hz, 3H), 1.60 – 1.47 (m, 4H), 1.39 – 1.13 (m, 22H), 0.86 (t, J = 7.3 Hz, 3H), 0.80 (t, J = 6.5 Hz, 3H).

<sup>13</sup>C-NMR δ 173.86, 129.98, 129.75, 64.08, 34.41, 32.01, 30.81, 29.85, 29.76, 29.75, 29.62, 29.41, 29.25, 29.21, 29.18, 27.28, 27.23, 25.08, 22.76, 19.23, 14.13, 13.74.

**Table 1. <sup>1</sup>H-NMR analysis of the fractions obtained by vacuum distillation**

	[CH=CH] UFA-PUFA	[CH <sub>2</sub> COOH] α-hydrogens <sup>a</sup>	[CH <sub>2</sub> CH=CH] UFA	[CH <sub>2</sub> CH <sub>2</sub> COOH] β-hydrogens <sup>c</sup>	[(CH <sub>2</sub> ) <sub>n</sub> ] C-chain <sup>c</sup>	[CH <sub>3</sub> ] ω-hydrogens
Forerun	1.90	2.00	3.38	2.22	21.23	3.05
Heart	2.03	2.00	3.92	2.33	20.82	3.02
Tail <sup>b</sup>	1.78	2.00	3.50	2.50	23.58	3.72
Dunder <sup>b</sup>	1.09	2.00	2.60	3.66	24.29	3.36
Theor. <sup>d</sup>	2.00	2.00	4.00	2.00	20.00	3.00

<sup>a</sup> Used as reference (2.00 protons). Assignments and ratio determined, as previously described [14].

<sup>b</sup> <sup>1</sup>H-NMR shows two groups of signals in the olefin region, due to the presence of PUFA.

<sup>c</sup> Signals partially overlapped.

<sup>d</sup> Theoretical, for oleic acid.

FT-IR (KBr,  $\text{cm}^{-1}$ ):  $\nu_{\text{C-H}}$  2925,  $\nu_{\text{C=O}}$  1738,  $\nu_{\text{C=C}}$  1655,  $\delta_{\text{C-O}}$  1243, 1175, 1064.

## 2.4 Reduction [18,19]

In a 1 L three-necked flask, fitted with two efficient condensers, 300 mL of dry butanol, 50 g (0.15 mol) of butyl oleate and 18 g (0.8 mol) of sodium metal chopped into small cubic pieces, were added. The reaction is slow at first, but soon it becomes more and more vigorous, the temperature easily reaching the boiling point of butanol, so care must be taken to not overheat the system. After a while (10-15 minutes) the temperature is decreased, and once that happens, some heating can be applied in order to keep a gentle reflux, which must be kept until all the sodium is consumed. The system is allowed to cool down to room temperature, and 100 mL of water are slowly and cautiously added through the top of one of the condensers. The reaction is gently refluxed again for an additional hour. The contents of the flask were poured into a beaker containing 500 mL of water, and stirred until both phases were separated. The organic phase was washed repeatedly with water and dried over anhydrous sodium sulfate. The remaining butanol was removed by distillation at atmospheric pressure, and could be re-used in the previous step. The resulting residue of oleyl alcohol was distilled in a Kugelrohr apparatus (0.01 mm Hg), to give 35 g (0.13 mol), 88% yield.

$^1\text{H-NMR}$  ( $\text{C}_6\text{D}_6$ )  $\delta$  5.34 (q, J = 4.8, 4.0 Hz, 2H), 4.27 (s, 1H), 3.62 – 3.41 (m, 2H), 2.15 – 1.87 (m, 4H), 1.58 – 1.42 (m, 4H), 1.26 (d, J = 17.7 Hz, 20H), 0.88 – 0.83 (m, 3H).

$^{13}\text{C-NMR}$  ( $\text{C}_6\text{D}_6$ )  $\delta$  130.19, 62.50, 33.31, 32.48, 30.36, 30.29, 30.22, 30.19, 30.14, 29.99, 29.93, 29.89, 27.78, 27.75, 26.49, 23.22, 14.47.

FT-IR (KBr,  $\text{cm}^{-1}$ ):  $\nu_{\text{O-H}}$  3331,  $\nu_{\text{C-H}}$  2924,  $\nu_{\text{C=C}}$  1654.  $\delta_{\text{C-O}}$  1464, 1057.

## 2.5 Synthesis of Bromotriphenylphosphine Bromide [20]

A stoichiometric amount of bromine (25 g, 156 mmol) was added dropwise over triphenylphosphine (40 g, 153 mmol) in 200 mL of a 4:1 mixture of dry diethyl ether and dry dichloroethane. Bromotriphenylphosphine bromide precipitated, and was isolated by filtration as a pale yellow solid, 51 g (0.12 mol), 79% yield.

$^1\text{H NMR}$   $\delta$  7.71 (t, J = 5.7 Hz, 1H), 7.59 – 7.50 (m, 4H).

$^{13}\text{C NMR}$   $\delta$  134.37 (d, J = 2.8 Hz), 132.38 (d, J = 11.1 Hz), 129.58 (d, J = 13.1 Hz), 126.04 (d, J = 108.2 Hz).

$^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  43.32 (s).

FT-IR (KBr,  $\text{cm}^{-1}$ ):  $\nu_{\text{C-H}}$  2989,  $\nu_{\text{C=C}}$  1616,  $\nu_{\text{C-Br}}$  535.

m.p.: 235°C (dec.)

## 2.6 Bromination [21,22]

To a solution of 10 g (37 mmol) of oleyl alcohol and 5 g (63 mmol) of pyridine in 100 mL of acetonitrile, 20 g (47 mmol) of bromotriphenylphosphine bromide was slowly added at 0°C. After the addition was completed (about 10 minutes), the ice bath was removed, the reaction allowed to warm to room temperature, and stirred for an additional hour. The mixture was then filtered through a short column of silica gel using a 1:10 ether-pentane mixture as eluent. The filtrate was concentrated in a rotary evaporator, to obtain oleyl bromide, 8 g (24 mmol), 66% yield.

$^1\text{H-NMR}$  (Acetone- $d_6$ )  $\delta$  5.33 (t, J = 4.7 Hz, 2H), 3.51 (t, J = 6.3 Hz, 2H), 2.02 (q, J = 6.2 Hz, 4H), 1.49 (p, J = 6.4 Hz, 2H), 1.37 – 1.23 (m, 22H), 0.86 (t, J = 6.6 Hz, 3H).

$^{13}\text{C-NMR}$  (Acetone- $d_6$ )  $\delta$  130.78, 34.06, 32.91, 30.76, 30.71, 30.57, 30.52, 30.34, 30.27, 30.14, 29.95, 29.76, 28.11, 28.09, 26.99, 23.62, 14.71.

FT-IR (KBr,  $\text{cm}^{-1}$ ):  $\nu_{\text{C-H}}$  3006,  $\nu_{\text{C=C}}$  1628,  $\nu_{\text{C-Br}}$  540.

## 3. RESULTS AND DISCUSSION

The ultimate goal of this work was to access the final product keeping all costs as low as possible. The commercial olive oil was chosen just because it was the cheapest available in the closest local supermarket. As revealed by direct  $^1\text{H-NMR}$  analysis, our olive oil contained approximately 80% of oleic acid esters [14].

For convenience, and as a way to get rid of minor components, like saturated fatty acids, gums, etc., we submitted the commercial oil to a saponification, followed by a Fischer esterification. After some trial and error, we

realized that butyl esters were the most convenient, as they turned up easier to handle, had less tendency to foam, gave cleaner products, and butanol was easier to recover, dry and recycle, thanks to its low miscibility with water. Also, butanol is much easier to dry, as compared with other lighter alcohols [23], because it can be pre-dried over solid magnesium sulfate, sodium carbonate, calcium oxide, or sodium hydroxide, followed by a simple distillation discarding a good 20-25% in volume of the head fraction, as the azeotrope with water (which contains about 37% water) is more volatile than the core distillate. For the use given in this step that should suffice but, if the anhydrous alcohol is desired, it can then be refluxed with sodium borohydride (about 1 g/liter of alcohol), followed by distillation from some sodium metal (about 1 g per liter of pre-dried solvent) [24].

The butyl esters so obtained were submitted to a Bouveault–Blanc reduction, in conditions similar to those previously described for the ethyl ester [19], using sodium in anhydrous butanol, followed by treatment with bromotriphenylphosphonium bromide to yield the corresponding alkyl bromide, also leaving the Z-stereochemistry and the C=C intact.

The purity of the oleyl alcohol and the oleyl bromide was estimated over 95% under the basis of their NMR spectra, and as such the product can be used for preparations and further synthetic elaboration.

#### 4. CONCLUSION

Pure oleyl bromide was commercially available (Sigma-Aldrich), but recently it was discontinued. Other sources also offer the product [25], but it is expensive (starting at U\$S 600 per gram). From an economical point of view, this simple preparation was much cheaper than the material sourced from the previously mentioned suppliers. The final overall yield obtained was of about 30%. All solvents used could be mostly recovered and re-cycled very efficiently.

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#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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