

# Studies on the Synthesis, Physical Properties, and Stability of Benzyl Ethers as Potential Heat Transfer Fluids †

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## Abstract

Heat transfer fluids (HTFs) play a crucial role across a range of industries, absorbing and conveying thermal energy in applications, including as essential components in concentrating solar power systems. As a continuation of previous work, we look at extended dibenzyl ethers as HTFs. The physical properties heat capacity and temperature-dependent density of selected dibenzyl ethers are investigated, and values compared with computational data using incremental methods for the estimation of group contributions within the structures towards the macroscopic properties of the compounds. Thermal stability of the dibenzyl ethers and their oxidative stability over longer period of time are studied, also.

**Keywords:** benzyl ethers; heat transfer fluid; heat capacity; density; oxidative degradation

## 1. Introduction [1]

A heat transfer fluid (HTF) is a gas or liquid that facilitates heat transfer by acting as an intermediary – absorbing heat in one part of a process (cooling), transporting and storing thermal energy, and releasing it in another part of the process (heating). The choice of HTF is largely determined by the specific process and system design, with the primary goal being to maximize heat transfer efficiency while minimizing energy input and overall cost [2]. HTFs and thermal oils used in high-temperature applications differ in their chemical composition; many are synthetic and can originate from either organic or inorganic sources, depending on the application requirements.

Organic HTFs encompass a wide range of compounds, including esters, diesters, polyglycols, water-glycol mixtures, paraffins, and waxes. Silicone-based greases and oils are also commonly used. For systems operating at elevated temperatures, inorganic fluids such as liquid sodium [3] (e.g., in nuclear reactors and in concentrated solar thermal energy systems) and molten nitrate salts (sodium nitrate–potassium nitrate mixtures [4]) are employed. More recently, advanced materials like carbon nanofibers, carbon nanotubes, and metallic nanopowders have been explored as potential HTFs [2].

Historically, most organic HTFs derived from aromatic or aliphatic petrochemicals were used within a temperature range of 0 to 350 °C. At higher temperatures, these

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compounds are prone to decomposition or oxidation [5]. Nevertheless, synthetic organic HTFs have regained interest due to their superior thermal properties, despite their higher cost.

HTFs and thermal oils are characterized by differences in kinematic viscosity, operating temperature range, and critical points such as pour point, boiling point, and flash point. Key performance parameters also include thermal energy storage capacity per unit volume, toxicity, and usable temperature range [6]. In evaluating an HTF, essential properties include high heat capacity and thermal conductivity, high flash point, wide operating temperature range (low melting and high boiling points), low viscosity and density, and high vapor pressure [7].

Environmental concerns have also been raised regarding HTFs [8]. Some possess relatively poor thermal performance compared to water. At room temperature, many HTFs exhibit higher viscosity, lower density, and reduced specific heat capacity and thermal conductivity. To compensate, they are often used at higher flow rates, requiring more pumping power to transfer the same amount of energy as water.

The authors have been investigating suitable organic HTFs for application in a newly developed solar thermoelectric generator [9]. This interest aligns with their ongoing work on ether synthesis [10], leading to the development of several new aryl ethers, particularly benzyl ethers, as potential HTFs. The current study focuses on this effort. Preliminary modeling indicated that these molecules may offer relatively high heat capacity. However, from the outset, the limited thermal stability of benzyl ethers was identified as a potential limitation.

The initial phase of research focused on the synthesis of dibenzyl ethers, supported by a graduation project that modeled the scale-up of production to an industrial level [11]. Subsequently, the scope was expanded to include the synthesis of oligobenzyl ethers, with the understanding that future work would need to establish more scalable production routes if these compounds were to be used as commercial HTFs.

Even 23 years after Metzger's influential article on chemical sustainability and 33 years after the Rio Earth Summit [12], efficient use of chemical resources remains essential. Purification steps often consume more solvent than the reactions themselves and involve solid-phase materials such as silica gel. However, while solid-phase materials can often be recycled, solvents present greater challenges. From this sustainability perspective, the authors explored solvent reduction strategies and investigated one-pot syntheses, including combined etherification and Suzuki cross-coupling reactions, in some cases performed under solvent-free conditions.

In the current contribution, we extend the scope of benzyl ethers that can be prepared under these conditions. Furthermore, the authors investigate the physical properties such as the heat capacity and the temperature-dependent density of selected dibenzyl ethers, and compare the values with computational data using incremental methods for the estimation of group contributions within the structures towards the macroscopic properties of the compounds. Finally, it was realized that while some of the dibenzyl ethers were stable at higher temperatures, some of the studied benzyl ethers were found to degrade oxidatively in air at rt over longer time periods. Possible mechanisms for these degradative transformations are discussed.

## 2. Experimental

**General.**—Melting points were measured with a Stuart SMP10 melting point apparatus and are un-corrected.  $^1\text{H}$  NMR (at 400 MHz) and  $^{13}\text{C}$  NMR (at 100.5 MHz) spectra were taken on a Varian 400 MHz spectrometer. IR measurements were performed on a Thermo Nicolet FT-IR spectrometer, model Nexus 470. Density measurements were performed on an Anton-Barr DMA 4500M densitometer. TGA measurements were carried

out with a Shimadzu TGA-50. Column chromatography was executed on silica gel S (Riedel de Haen) and on silica gel (Merck grade 9385).

**Stability tests for compounds of the 4-bromobenzyl series.**—4-Bromobenzyl *n*-pentyl ether (4.19 g, 16.3 mmol) was kept at ambient temperature exposed to air for 12 months. The sample started showing a precipitate after 60 days. After 12 months, the sample was separated by column chromatography on silica gel (hexane/ether/CH<sub>2</sub>Cl<sub>2</sub> 4:1:1) after filtering off the precipitate and analysed to contain 4-bromobenzoic acid (**10a**, 1.44 g, 43.9%), 4-bromobenzaldehyde (**9a**, 150 mg, 5.0%), and *n*-pentyl 4-bromobenzoate (**11a**, 846 mg, 19.1%): 4-bromobenzoic acid (**10a**) as a colorless needles, mp. 252 °C (Lit. 252–254 °C):  $\nu_{\max}$  (KBr/cm<sup>-1</sup>) 3420–2050 (bs, OH), 1674, 1567, 1425, 1319, 1296, 1278, 1176, 1127, 1108, 1068, 1011, 927, 849, 756;  $\delta_{\text{H}}$  (400 MHz, DMSO-d<sub>6</sub>) 7.65 (2H, d, <sup>3</sup>J = 8.4 Hz), 7.82 (2H, d, <sup>3</sup>J = 8.4 Hz);  $\delta_{\text{C}}$  (100.5 MHz, CDCl<sub>3</sub>) 131.0 (C<sub>quat</sub>), 131.6 (2C, CH), 131.8 (2C, CH), 132.4 (C<sub>quat</sub>), 168.2 (C<sub>quat</sub>, CO);  $\delta_{\text{C}}$  (100.5 MHz, DMSO-d<sub>6</sub>) 127.3 (C<sub>quat</sub>), 130.4 (C<sub>quat</sub>), 131.7 (2C, CH), 132.1 (2C, CH), 167.1 (C<sub>quat</sub>); 4-bromobenzaldehyde (**9a**) as a colorless solid, mp. 61 °C;  $\nu_{\max}$  (KBr/cm<sup>-1</sup>) 2859, 1698, 1588, 1573, 1382, 1204, 1065, 1009, 834, 811;  $\delta_{\text{H}}$  (400 MHz, CDCl<sub>3</sub>) 7.67–7.73 (4H, m), 9.96 (1H, s, CHO);  $\delta_{\text{C}}$  (100.5 MHz, CDCl<sub>3</sub>) 129.8 (C<sub>quat</sub>), 131.0 (2C, CH), 132.4 (2C, CH), 135.0 (C<sub>quat</sub>), 191.1 (CHO), and *n*-pentyl 4-bromobenzoate (**11a**) [50] as a colorless oil;  $\nu_{\max}$  (KBr/cm<sup>-1</sup>) 2957, 2932, 2871, 1723, 1591, 1484, 1467, 1397, 1271, 1173, 1103, 1069, 1012, 848, 757, 683;  $\delta_{\text{H}}$  (400 MHz, CDCl<sub>3</sub>) 0.90 (3H, t, <sup>3</sup>J = 6.4 Hz, CH<sub>3</sub>), 1.32–1.39 (4H, m), 1.73–1.77 (2H, m), 4.29 (2H, t, <sup>3</sup>J = 6.4 Hz, OCH<sub>2</sub>) 7.56 (2H, d, <sup>3</sup>J = 8.8 Hz), 7.88 (2H, d, <sup>3</sup>J = 8.8 Hz);  $\delta_{\text{C}}$  (100.5 MHz, CDCl<sub>3</sub>) 14.0 (CH<sub>3</sub>), 22.3 (CH<sub>2</sub>), 28.2 (CH<sub>2</sub>), 28.4 (CH<sub>2</sub>), 65.4 (OCH<sub>2</sub>), 127.9 (C<sub>quat</sub>), 129.4 (C<sub>quat</sub>), 131.1 (2C, CH), 131.6 (2C, CH), 165.9 (C<sub>quat</sub>, CO).

Similarly, for 4-bromobenzyl *n*-butyl ether was found: *n*-butyl 4-bromobenzoate:  $\nu_{\max}$  (neat/cm<sup>-1</sup>) 1721;  $\delta_{\text{H}}$  (400 MHz, CDCl<sub>3</sub>) 0.97 (3H, t, <sup>3</sup>J = 7.2 Hz), 1.42–1.51 (2H, m), 1.70–1.77 (2H, m), 4.31 (2H, t, <sup>3</sup>J = 6.4 Hz), 7.56 (2H, d, <sup>3</sup>J = 8.8 Hz), 7.89 (2H, d, <sup>3</sup>J = 8.8 Hz);  $\delta_{\text{C}}$  (100.5 MHz, CDCl<sub>3</sub>) 13.8 (CH<sub>3</sub>), 19.2 (CH<sub>2</sub>), 30.7 (CH<sub>2</sub>), 65.1 (OCH<sub>2</sub>), 127.9 (C<sub>quat</sub>), 129.4 (C<sub>quat</sub>), 131.1 (2C, CH), 131.6 (2C, CH), 166.0 (C<sub>quat</sub>, CO).

For 4-bromo benzyl *n*-hexyl ether was found: *n*-hexyl 4-bromobenzoate:  $\nu_{\max}$  (neat/cm<sup>-1</sup>) 1722;  $\delta_{\text{H}}$  (400 MHz, CDCl<sub>3</sub>) 0.89 (3H, t, <sup>3</sup>J = 6.8 Hz), 1.66–1.77 (2H, m), 1.30–1.44 (6H, m), 4.29 (2H, t, <sup>3</sup>J = Hz, OCH<sub>2</sub>), 7.55 (2H, d, <sup>3</sup>J = 8.4 Hz), 7.88 (2H, d, <sup>3</sup>J = 8.4 Hz);  $\delta_{\text{C}}$  (100.5 MHz, CDCl<sub>3</sub>) 14.0 (CH<sub>3</sub>), 25.5 (CH<sub>2</sub>), 25.7 (CH<sub>2</sub>), 28.6 (CH<sub>2</sub>), 31.4 (CH<sub>2</sub>), 65.4 (OCH<sub>2</sub>), 127.9 (C<sub>quat</sub>), 129.4 (C<sub>quat</sub>), 131.1 (2C, CH), 131.6 (2C, CH), 165.9 (C<sub>quat</sub>, CO).

### 3. Results and Discussion

#### 3.1. Typical Synthesis of the Studied Dibenzyl Ethers

Non-symmetric ethers are commonly prepared by Williamson ether synthesis [13]. Here, the authors prepared simple dibenzyl ethers by reaction benzyl bromides and chlorides with benzyl alcohols using powdered KOH as base and DMSO as solvent. In order to circumvent DMSO in the work-up of the reaction and for subsequent reactions where Suzuki-cross coupling was added as a concurrent transformation, the authors changed to solventless systems (Scheme 1) under phase transfer catalysis (PTC), following a procedure analogous to Rao and Senthilkumar [14], utilizing finely KOH as base and benzyltributylammonium chloride (BTBAC) as PTC. Double Williamson ether syntheses as shown in Scheme 2 lead to extended benzyl ether systems. These syntheses can be combined with Suzuki coupling reactions in one pot (Scheme 3). When one utilizes formylphenylboronic acids such as **6**, the formyl group in product **7** can be reduced to an alcohol function (NaBH<sub>4</sub>) and converted to a bromide (PBr<sub>3</sub>), making these derivatives of **7** precursors of a subsequent Williamson ether synthesis to larger benzyl ethers [1].



$$C_p^l(T) = C_{p0}^l(T) + \sum_i N_i C_{p1-i}^l(T) + w \sum_j M_j C_{p2-j}^l(T) + z \sum_k O_k C_{p3-k}^l(T) \quad (1)$$

with

$$C_{p\ q\ q^{th}level-i,j,or\ k}^l(T) = a_{q-i,j,or\ k} + b_{q-i,j,or\ k} \left(\frac{T}{100}\right) + d_{q-i,j,or\ k} \left(\frac{T}{100}\right)^2 \quad (2)$$

where in Equation (1):  $C_{p1-i}^l(T)$  is the contribution of the first-level group of type  $i$ ,  $C_{p2-j}^l(T)$  is the contribution of the second-level group of type  $j$ , and  $C_{p3-k}^l(T)$  is the contribution of the third-level group of type  $k$ .  $N_i$ ,  $M_j$ , and  $O_k$  indicate the number of occurrences of the individual groups (of type  $i$ ,  $j$ , or  $k$ , respectively) in a compound.  $C_{p0}^l(T)$  (which could be considered as the contribution of the zero-level group) is an additional adjustable parameter. Variables  $w$  and  $z$  are weighting factors that are assigned 0 or 1, depending on whether the second-level and third-level contributions, respectively, are used or not. In Equation (2),  $a_{q-i,j,or\ k}$ ,  $b_{q-i,j,or\ k}$ , and  $d_{q-i,j,or\ k}$  are adjustable parameters for the temperature dependence of  $C_{p0}^l(T)$ ,  $C_{p1-i}^l(T)$ ,  $C_{p2-j}^l(T)$ , and  $C_{p3-k}^l(T)$ .

Within this method, there are two approaches (Nonhierarchical (NH) and Hierarchic (H)), which differ from each other by only the adjustable parameters ( $a_{q-i,j,or\ k}$ ,  $b_{q-i,j,or\ k}$ , and  $d_{q-i,j,or\ k}$ ). The other group estimation method used, namely the method of Joback and Reid [15], is a first order approximation unlike the previous method. It is applied through Equation (3):

$$C_p^0(T) = \sum (a) - 37.93 + \left[ \sum (b) + 0.201 \right] T + \left[ \sum (c) - 3.91 \times 10^{-4} \right] T^2 + \left[ \sum (d) + 2.06 \times 10^{-7} \right] T^3 \quad (3)$$

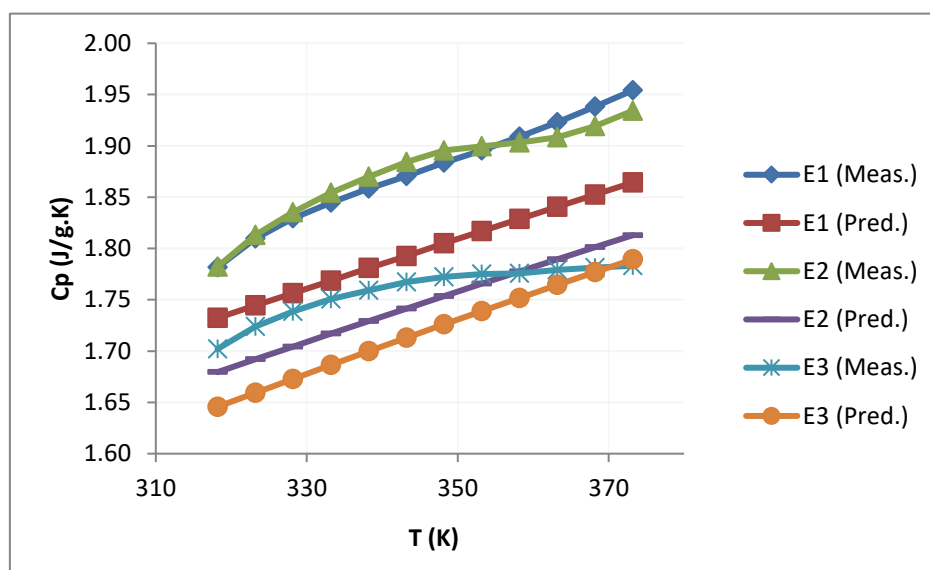
where,  $C_p^0(T)$ : ideal gas heat capacity (J/mole.K). T: temperature (K). a, b, c and d: adjustable parameters.  $\Sigma$ : indicates that, for the particular property of interest, one sums the product of the number of times a group appears in the compound and the group contributions.

The measured and estimated heat capacities  $C_p$  [J/g.K] (J/mole.K) for ethers benzyl phenylethyl ether (E1), 2-benzyloxy dibenzyl ether (E2) and 2-benzyloxymethyl-4'-(4-methoxybenzyloxymethyl)biphenyl (E3) at three different temperatures are shown in Table 1.

From Table 1, it is obvious that predicted heat capacities of the specified ethers (E1, E2 and E3) by Kolská's method [22] are much closer the measured values compared to Joback and Reid's approach. This can be justified that Kolská's method [22], as recently published, depends on three level group contribution analysis which covers the mono-functional molecules' contribution in groups of the first level, while the groups of the second and third levels improve the more-complex prediction of compounds. In addition, it relays on higher number of compounds with known properties. At the same time, the Hierarchic (H) approach of Kolská's method [22] is more reliable than the Nonhierarchical (NH) approach, especially with structurally complicated ethers (E2 and E3). Noting that, there was an average error of less than 0.5% between experimentally measured [23] and predicted (through H Approach) heat capacities of Dibenzyl ether over the studied temperatures' range, unlike with (NH) approach (average error of 23%). Similarly with diphenyl ether [24], the average error was less than 3% considering the (H) approach vs. 12% with (NH) approach. Thus, the measured heat capacities and predicted values by the (H) approach of Kolská method [22] for ethers (E1–E3) at different temperatures are shown in Figure 1.

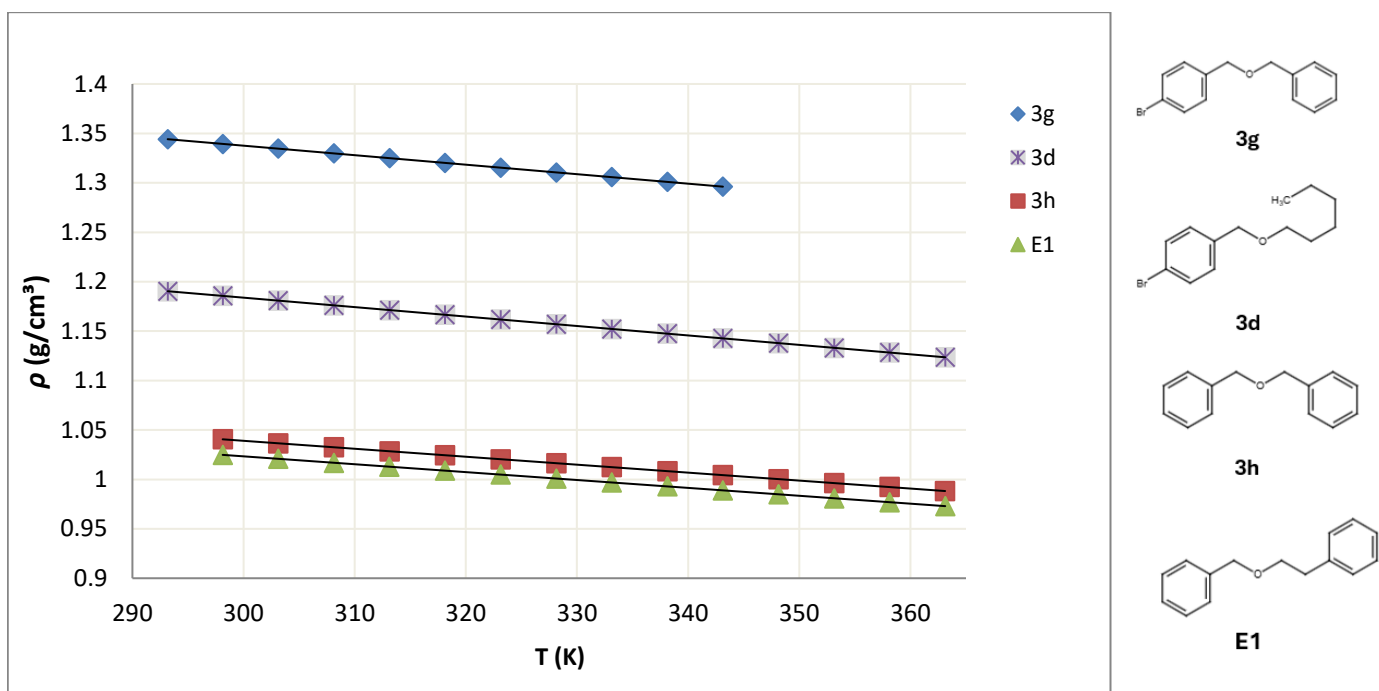
**Table 1.** Measured and estimated heat capacities for ethers E1, E2 and E3 at three different temperatures.

Compound	T (K)	Cp [J/g·K] (J/mole·K)			
		Kolská et al. [22]		Joback & Reid [15]	Measured
		(NH) Approach	(H) Approach		
E1	323	1.84 (391.1)	1.74 (370.3)	1.28 (271.0)	1.81 (384.2)
	348	1.89 (400.3)	1.80 (383.1)	1.37 (290.5)	1.88 (399.9)
	373	1.92 (407.3)	1.86 (495.8)	1.46 (309.4)	1.95 (414.8)
E2	323	2.13 (678.9)	1.69 (538.7)	1.26 (402.6)	1.81 (577.4)
	348	2.09 (666.9)	1.75 (558.3)	1.35 (430.5)	1.90 (603.5)
	373	2.05 (653.6)	1.81 (577.2)	1.44 (457.5)	1.93 (615.9)
E3	323	2.27 (963.6)	1.66 (704.5)	1.27 (538.4)	1.72 (731.8)
	348	2.19 (929.0)	1.73 (732.7)	1.35 (574.3)	1.77 (752.4)
	373	2.09 (888.4)	1.79 (759.6)	1.43 (609.1)	1.78 (757.0)

**Figure 1.** Measured (Meas.) and predicted (Pred.) heat capacities of ethers (E1–E3).

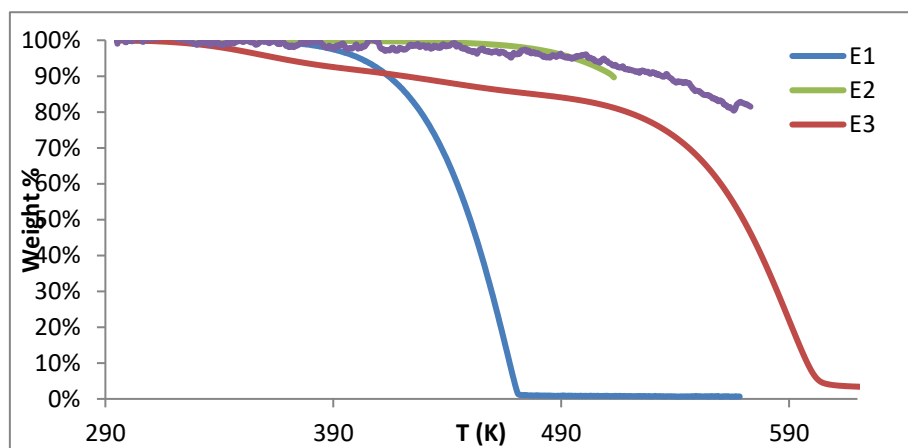
From Figure 1, one can realize that it is not necessary for the more complicated ethers to have higher specific heat capacities (per mass basis). This can be justified that the simple structures (e.g., E1) have the potential to form stronger intermolecular bonding compared to complicated structures (e.g., E2 & E3).

Density measurements of four ethers (**3d**, **3g**, **3h**, and **E1**) were also performed at temperatures up to 363 K (90 °C) (see Figure 2). Interestingly, as can be noted from Figure 2, all of the four studied ethers exhibit an exact linear decrease in density with temperature. The bromo group in **3d** and **3g** makes their densities higher compared to **3h** and **E1**. Thus **3d** and **3g** share the same slope value (−0.001), while also **E3** and **E4** themselves share the same slope (−0.0008). Moreover, the benzyl-group (**3g**) leads to a higher density of molecules as compared to the hexyl-group (**3d**).

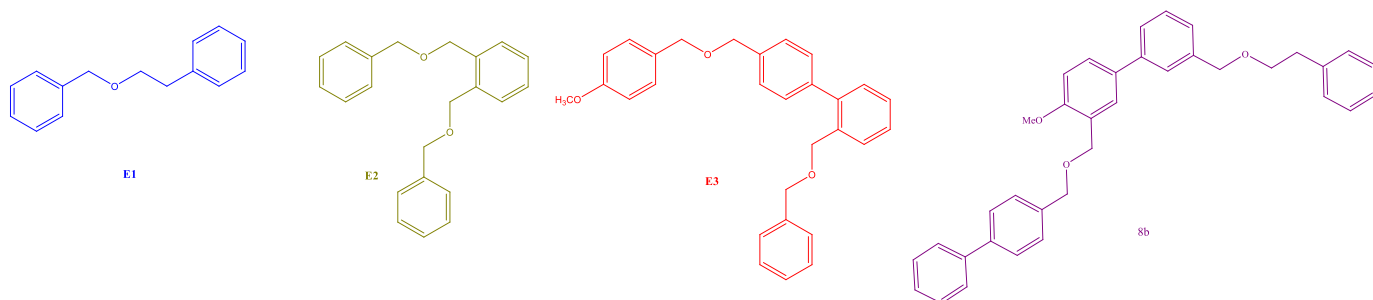


**Figure 2.** Density measurements of ethers 4-bromobenzyl benzyl ether (**3g**); 4-bromobenzyl hexyl ether (**3d**); dibenzyl ether (**3h**) and benzyl phenylethyl ether (**E1**).

Lastly, thermal gravimetric analysis (TGA) has shown that especially the extended ethers such as **8b** are stable up to 573 K (300 °C), even in air without large weight loss %. Figure 3, shows the thermal stability of four ethers (**E1**, **E2**, **E3** and **8b**).

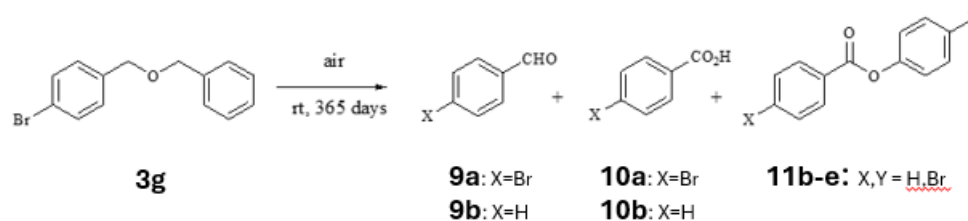


**Figure 3.** Thermal stability study of ethers (**E1**, **E2**, **E3** and **8b**), as weight% remaining vs. temperature (K), performed by TGA.









**Scheme 6.** Degradation of 4-bromobenzyl benzyl ether (**3g**) by autoxidation at rt open to ambient air for 1 year.

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