o-Benzenedisulfonimide as a Soft, Efficient, and Recyclable Catalyst for the Acylation of Alcohols, Phenols, and Thiols under Solvent-Free Conditions: Advantages and Limitations

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Abstract: *o*-Benzenedisulfonimide turns out to be a highly efficient Brønsted acid catalyst for the acylation of a number of alcohols, phenols, and thiols under a metal- and solvent-free procedure; reaction conditions are mild and yields very good. After the workup, the catalyst can be easily recovered and purified, ready to be reused, with economic and ecological advantages.

Key words: *o*-benzenedisulfonimide, acid catalysis, recyclable catalyst, acylation reaction

Functional group protection is often essential in organic synthesis, so many methods and procedures of both protection and deprotection are continuously proposed in the literature. Alcohols, phenols, and thiols are routinely protected by acylation and, despite the high number of known procedures, new and more efficient methodologies are still in demand. The most commonly used reagents are acid anhydrides, although in the reaction one acyl moiety is lost, and acetic or benzoic anhydrides are the most frequently employed. The reaction with primary and secondary alcohols normally requires the presence of a suitable catalyst, whereas a strong base catalyst is needed for hindered tertiary alcohols.[1]

Taking into consideration only the literature concerning the acylation of alcohols, phenols, thiols, and amines with acid anhydrides, the number of recent reports is astonishing, including the use of both homogeneous and heterogeneous catalysts. Most methods employ solvent-free conditions, room temperature (for acetylation with Ac_2O) or heating (for other anhydrides, less commonly used), in the presence of Brønsted or Lewis acid catalysts.

Various organic and inorganic catalysts have been employed. The highest number of references deal with the use of Lewis acids,[2] amongst them: $Mg(NTf_2)_2$,[3] $Mg(HSO_4)_2$,[4] $Al(OTf)_3$,[5] $Sc(OTf)_3$,[6] lithium salts,[7] distannoxane derivatives,[8] $Er(OTf)_3$,[9] $ErCl_3$,[10] $SbCl_3$,[11] $NbCl_5$,[12] $InCl_3$,[13] $TaCl_5$,[14] $CoCl_2$,[15] tin(IV) porphyrin derivatives,[16] $La(NO_3)_3$,[17] lanthanide(III) tosylates,[18] copper salts,[19] bismuth oxide perchlorate,[20] zirconium salts,[21] cerium salts,[22] a Mn(III) complex,[23] $Gd(OTf)_3$,[24] $Al(HSO_4)_3$,[25] $BF_3{\cdot}OEt_2$,[26] cobalt polyoxometalate,[27] bismuth salts,[28] and silica derivatives.[29]

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Protic acid catalysis is less common, both under homogeneous and heterogeneous conditions. 4-Toluenesulfonic acid (alcohols; toluene, reflux),[30] phosphomolybdic acid (alcohols, phenols, and amines; neat, r.t., long reaction times),[31] 3-nitrobenzeneboronic acid (alcohols; r.t.),[32] and sulfamic acid (alcohols and phenols)[33] have been used.

Under heterogeneous conditions, solid acids such as montmorillonite,[34] zeolites,[35] zirconia,[36] Nafion-H,[37] macroporous metal oxides,[38] solid superacid,[39] and heteropolyacids,[40] have also been exploited for acetylation or acylation (in a few cases) of alcohols, phenols, thiols, and amines, under solvent-free conditions and at reflux or room temperature. In other examples of heterogeneous catalysis for O- and N-acetylation, the protic acid catalyst is supported on a high-surface insoluble inorganic or organic support, such as sulfated zirconia[41a] or MoO_3[41b] on alumina, and $NaHSO_4$,[42a] phosphomolybdic acid,[42b] P_2O_5,[42c] sulfuric acid,[42d] phosphotungstic acid,[42e] or fluoroboric acid[42f] on silica gel, under solvent-free conditions at room temperature or reflux.

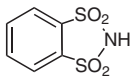
Ionic liquids have been used as the solvent and catalyst for the acetylation of alcohols and phenols at room temperature,[43] along with halogenated compounds[44] and onium salts[45] as acylation catalysts of alcohols and thiols, in dichloromethane or under solvent-free conditions, and at room temperature or with heating.

Furthermore, a few references describe solvent- and catalyst-free reactions, in particular the acylation of amines and arenethiols at room temperature,[46a] and the acylation of alcohols, amines, and thiols at 80–85 °C,[46b] or microwave induced[46c] at room temperature.

The high number of reported methodologies illustrates the importance of the development of new simple, low-cost, and environmentally benign procedures, involving solvent- and/or metal-free recyclable catalytic systems.

Recently, we reported the use of *o*-benzenedisulfonimide (**1**, Figure 1) as a new Brønsted acid organocatalyst, in some common acid-catalyzed organic reactions, such as the synthesis of ethers, esters, and acetals, acetal cleavage and interconversion, and pinacol rearrangement.[47]

For the synthesis of **1**, described for the first time in 1921[48] and 1926,[49] and more recently with modified procedures,[50–52] the key intermediate is *o*-benzenedisulfonyl chloride, which can be prepared starting from *o*-benzenedisulfonic acid dipotassium salt,[49,52] anthranilic acid,[53] 2-

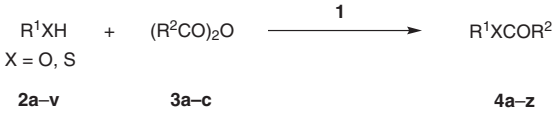


1

Figure 1

aminobenzenesulfonic acid,[48–51,54] and 1,2-bis(methylsulfanyl)benzene.[55] Now, both *o*-benzenedisulfonyl chloride and **1** are commercially available.

In this paper, we wish to report a new application of sulfonimide **1** as a nontoxic, nonvolatile, and noncorrosive recyclable catalyst, in an alternative, advantageous method for the solvent- and metal-free acylation of alcohols, phenols, and thiols with acid anhydrides (Scheme 1). Amines were not reacted because, as well-known and reported in the literature, their acylation with acid anhydrides does not require use of any catalyst.[46a]



Scheme 1

Further valuable aspects of the new procedure are the possibility of easy recovery of **1** in high yield from the reaction mixture, due to its complete solubility in water, and its reuse without loss of catalytic activity in further reactions, with economic and ecological advantages.

To assess the generality of the method, we investigated the scope and limitations of the reaction by treating various alcohols, phenols, and thiols **2a**–**v**, with acetic anhydride (**3a**), propanoic anhydride (**3b**), and benzoic anhydride (**3c**). Under the optimized conditions, the reactions were very mild: nearly equimolar amounts of reagents (**2**/**3**, 1:1.1), low catalytic load (**1**, 5.0 mol%), very short reaction times (5 min, unless otherwise specified), and room temperature for the acylation with acetic or propanoic anhydrides, whereas the benzoylation required heating to 60 or 80 °C. Complete conversion and good yields were generally obtained. Table 1 lists the substrates considered for acylation **2a**–**v**, the acid anhydrides employed **3a**–**c** and the acylation products **4a**–**z** that were isolated and purified by flash column chromatography.

Acetylation proceeded with high yields of pure acylation products, starting both from phenols **2a** and **2b** (entries 1 and 4), even sterically hindered as **2d**, (entry 6), and from aliphatic and benzylic primary, secondary and tertiary alcohols such as **2e** and **2g** (entries 7 and 12), **2m**, **2q**, and **2r** (entries 20, 24, and 26), and **2s** and **2t** (entries 27 and 29). High yields were also obtained starting from the sterically hindered phenol **2d** and secondary or tertiary alcohols **2r**, **2s**, and **2t**, although in the last example the reagent ratio **2t**/**3a** was 1:2. The reaction worked well also with primary and secondary allylic alcohols, **2i**–**k** and **2n**,**o**, affording the acylation products in good yields and with complete stereoselectivity without isomerization or formation of undesired byproducts (entries 16–18 and entries 21, 22). Furthermore, no racemization was observed upon reaction of the chiral substrate **2m** (entry 20).

However, the reaction failed in a few cases. A mixture of acid-catalyzed isomerization products, acylated or not, was recovered from the acetylation of linalol (**2l**) (entry 19) and 1-phenylprop-2-en-1-ol (**2p**) (entry 23), a mixture of olefinic products derived from competitive side reactions of elimination and dimerization via a carbocationic intermediate were isolated from the tertiary alcohol **2u** (entry 31), while with triphenylmethanol the reaction did not proceed, probably stopped at the conversion of **2v** into the stabilized intermediate triphenylmethyl carbocation.

The reaction also gave excellent results in a large-scale preparation starting from **2e** (entry 7, 0.1 mol, yield% in parentheses).

The optimized reaction conditions for the O-acetylation were then applied to the S-acylation of thiophenol (**2c**) (entry 5), octane-1-thiol (**2f**) (entry 10), and phenylmethanethiol (**2h**) (entry 15) with very good results in short reaction times.

To further explore the applicability of the proposed method, some substrates were acylated by propanoic anhydride (**3b**); carrying out the reactions under the same conditions (reagent ratio, 5 mol% catalyst, temperature, and reaction time), we obtained comparable, good yields of pure acylated products **4b**,**l**,**v** (entries 2, 13, and 25).

Finally, we carried out some O- and S-acylations with benzoic anhydride (**3c**). Accordingly to literature reports, the reaction proceeded with success only with heating of the reagents at 60 °C or 80 °C, in the presence of *o*-benzenedisulfonimide (5 mol%) as catalyst. The reaction was applied to 1-naphthol (**2a**, entry 3, 87% yield), octan-1-ol (**2e**, entries 8 and 9, at 60 °C or 80 °C, 70% and 81% yields respectively; higher temperatures, shorter reaction times, and improved yield), phenylmethanol (**2g**, entry 14, 91%), the hindered 1,1,1-trichloro-2-methylpropan-2-ol (**2t**, entry 30, reagent ratio **2t**/**3c**, 1:2; 71%) and octane-1-thiol (**2f**, entry 11, 82%).

Quite surprisingly, the reaction failed with 1-phenyl-2-methylpropan-2-ol (**2s**); in this case, heating the reaction mixture favored dehydrative elimination over benzoylation (entry 28), whereas acetylation run at room temperature gave 93% of acylated product **4x** (entry 27).

In Table 1 we reported the yields of the same products obtained in the Brønsted acid catalyzed acylation starting from the corresponding alcohols or phenols and acid anhydrides. Our yields are comparable or better, considering that our conditions are milder than those in the literature. For instance, more than one equivalent of anhydride for each hydroxy group,[32,33,42b,d,e] halogenated solvent,[33,42a,d,e] and/or longer reaction times[42f] were required.

In conclusion, we have described a new application of *o*-benzenedisulfonimide, a nontoxic, nonvolatile, and uncorrosive Brønsted acid, as new organocatalyst in an efficient, environmentally friendly, solvent-free acylation of alcohols, phenols, and thiols.

Table 1 *o*-Benzenedisulfonimide-Catalyzed Acylation of Alcohols, Phenols, and Thiols[a]

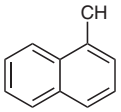
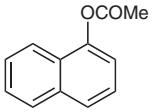
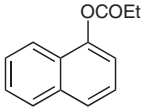
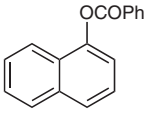
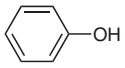
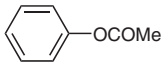
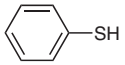
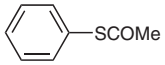
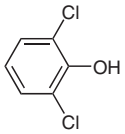
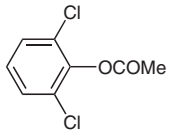
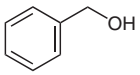
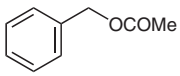
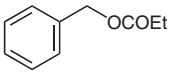
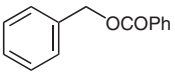
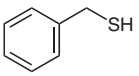
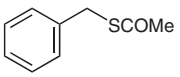
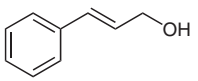
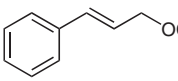
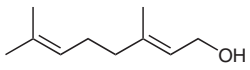
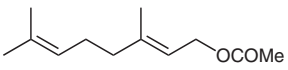
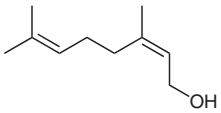
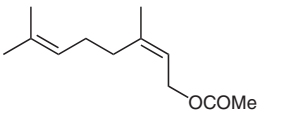
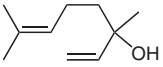
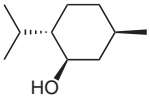
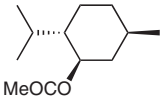
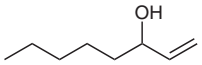
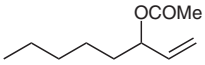
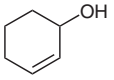
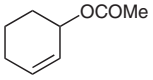
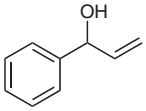
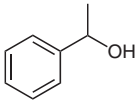
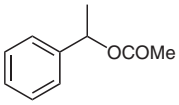
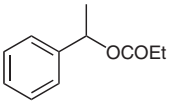
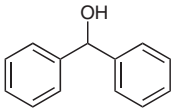
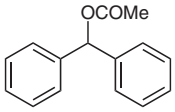
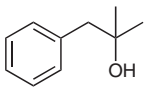
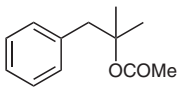
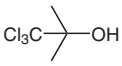
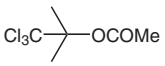
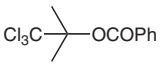
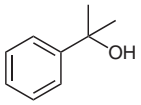
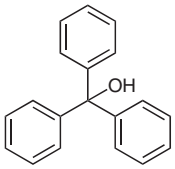
Entry	Substrate **2**	Anhydride **3**	Time (min)	Temp (°C)	Product **4**	Yield[b] (%)	Lit. yield[c] (%)
1	**2a** 	$(MeCO)_2O$ **3a**	5	r.t.	**4a** 	100	90,[42d,e] 91,[42f] 98[33]
2	**2a**	$(EtCO)_2O$ **3b**	5	r.t.	**4b** 	83	
3	**2a**	$(PhCO)_2O$ **3c**	2 h	60	**4c** 	87	
4	**2b** 	**3a**	5	r.t.	**4d** 	89	87,[42d] 89,[42e] 92,[42f] 94,[41a] 97,[31] 98[33]
5	**2c** 	**3a**	5	r.t.	**4e** 	100	
6	**2d** 	**3a**	4 h	r.t.	**4f** 	95	
7	**2e** $Me(CH_2)_6CH_2OH$	**3a**	5	r.t.	**4g** $Me(CH_2)_6CH_2OCOMe$	96 (98)[d]	88,[33] 90,[32,42d] 95[42e]
8	**2e**	**3c**	4 h	60	**4h** $Me(CH_2)_6CH_2OCOPh$	70	
9	**2e**	**3c**	1.5 h	80	**4h** $Me(CH_2)_6CH_2OCOPh$	81	
10	**2f** $Me(CH_2)_6CH_2SH$	**3a**	30	r.t.	**4i** $Me(CH_2)_6CH_2SCOMe$	88	
11	**2f**	**3c**	7 h	60	**4j** $Me(CH_2)_6CH_2SCOPh$	82	
12	**2g** 	**3a**	5	r.t.	**4k** 	98	90,[42d,71] 93,[41a] 94,[42f] 95,[31] 96,[42e] 99,[33]
13	**2g**	**3b**	5	r.t.	**4l** 	82	
14	**2g**	**3c**	1 h	80	**4m** 	91	
15	**2h** 	**3a**	30	r.t.	**4n** 	88	85[42f]
16	**2i** 	**3a**	5	r.t.	**4o** 	87	94,[31] 95[32]
17	**2j** 	**3a**	1	r.t.	**4p** 	84	96[31]
18	**2k** 	**3a**	10	r.t.	**4q** 	90	91[42f]
19	**2l** 	**3a**	–[e]	r.t.	–[e]	–[e]	
20	**2m** 	**3a**	10	r.t.	**4r** 	95	83,[42f] 94,[42a] 98[42b]

Table 1 *o*-Benzenedisulfonimide-Catalyzed Acylation of Alcohols, Phenols, and Thiols[a] (continued)

Entry	Substrate **2**	Anhydride **3**	Time (min)	Temp (°C)	Product **4**	Yield[b] (%)	Lit. yield[c] (%)
21	**2n** 	**3a**	5	r.t.	**4s** 	80	
22	**2o** 	**3a**	5	r.t.	**4t** 	80	
23	**2p** 	**3a**	–[e]	r.t.	–[e]	–[e]	
24	**2q** 	**3a**	5	r.t.	**4u** 	94	87,[42f] 93,[31] 97,[31]
25	**2q**	**3b**	15	r.t.	**4v** 	88	
26	**2r** 	**3a**	2 h	r.t.	**4w** 	78 (82)[f]	90,[42d] 95,[31] 98,[42e] 99[32]
27	**2s** 	**3a**	5	r.t.	**4x** 	93	
28	**2s**	**3c**	–[f]	80	–[g]	–[g]	
29	**2t** 	**3a**	1 h	r.t.	**4y** 	86[f]	
30	**2t**	**3c**	5 h	80	**4z** 	71[f]	
31	**2u** 	**3a**	–[h]	r.t.	–[h]	–[h]	
32	**2v** 	**3a**	–[i]	r.t.	–[i]	–[i]	

[a] Reaction conditions: **2/3** (1:1.1), *o*-benzenedisulfonimide (5 mol%).
[b] Yields refer to pure isolated products (flash chromatography; PE–Et_2O, 95:5).
[c] Literature yields refer to Brønsted acid catalyzed acylation reactions.
[d] Value in parentheses refers to scaled-up run (see experimental section).
[e] Under these conditions, GC-MS analyses showed the presence of acylated isomers in a mixture with other unidentified products. In entry 19, **4p** and **4q** were identified by comparison with the same pure products from entries 17 and 18. In entry 23, two peaks with *m*/*z* 176 [M^+]), attributable to the expected acylation product and to the isomerized cinnamyl derivative, were detected.
[f] Reaction conditions: **2/3** (1:2).
[g] Dehydration product was identified by GC-MS analyses (*m*/*z* 132 [M^+]).
[h] GC-MS analyses showed the presence of only traces of the expected ester (*m*/*z* 178 [M^+]); olefinic products were identified and isolated as major products: 2-phenylprop-1-ene [yield: 31%; *m*/*z* 118 [M^+]; ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ = 2.11 (d, *J* = 3.6 Hz, 3 H), 5.01–5.04 (m, 1 H), 5.31 (d, *J* = 3.4 Hz, 1 H), 7.18–7.33 (m, 3 H), 7.39–7.45 (m, 2 H)][56] and 4-methyl-2,4-diphenylpent-1-ene [yield: 45%; *m*/*z* 236 [M^+]; ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ = 1.17 (s, 6 H), 2.78 (s, 2 H), 4.73 (m, 1 H), 5.09 (d, *J* = 1.8 Hz, 1 H), 7.09–7.26 (m, 10 H)].[22a] They probably derive from competitive side reactions of alcohol **2u**.
[i] The reaction does not proceed. Disappearance of **2v** was observed, but only traces of acylated product were detected by GC-MS analysis (*m*/*z* 302 [M^+]). The reaction probably stops owing to the formation of the highly stabilized triphenylmethyl carbocation.

All the reactions were conducted in vials using analytical grade reagents and were monitored by GC and GC-MS spectrometry. GC-MS data were recorded with an HP 5989B mass selective detector connected to an HP 5890 GC cross-linked methyl silicone capillary column. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ with a Bruker Avance 200 spectrometer at 200 MHz and 50 MHz, respectively; chemical shifts are given relative to $CDCl_3$. TLC were performed on Merck silica plates GF 254; flash chromatography was carried out on silica gel (particle size 0.032–0.063 mm). Petroleum ether (PE) refers to the fraction boiling in the range 40–60 °C. Structure and purity of all the products were confirmed by comparison of their spectral data (MS and ^{1}H NMR) with those reported in literature or with those of available commercial samples. Commercially available reagents and solvents were purchased from Aldrich and were used without purification or distillation prior to use; Dowex 50X8 ion-exchange resin was purchased from Fluka. *o*-Benzenedisulfonimide (**1**) was prepared as described in the literature.[57]

Acylation; General Procedure

A mixture of substrate **2** (2.0 mmol), acid anhydride **3** (2.2 mmol, 1.1 equiv), and *o*-benzenedisulfonimide (**1**, 5 mol%, 0.1 mmol, 0.022 g) was stirred at r.t. in a vial. The exothermic reaction was stopped after 5 min, previous TLC and GC analyses. The mixture was treated with Et_2O and H_2O (1:1, 10 mL); organic layer was washed with 5% aq $NaHCO_3$, dried (Na_2SO_4), and concentrated under reduced pressure affording virtually pure products **4**, purified by flash chromatography on a short column (silica gel, PE–Et_2O, 9.5:0.5).

The aqueous layer from the various reactions were collected and evaporated under reduced pressure. The residue was passed through a column of Dowex 50X8 ion-exchange resin (1.6 g for 1 g of product), eluting with H_2O. After removal of H_2O under reduced pressure, virtually pure (^{1}H NMR) *o*-benzenedisulfonimide (**1**) was recovered; mp 192–194 °C (toluene) (Lit.[57] 192–194 °C).

In the case of entry 7, the reaction was carried out on a large scale, starting from alcohol **2e** (13.02 g, 0.1 mol) and Ac_2O (**3a**, 11.22 g, 0.11 mol), in the presence of 5 mol% *o*-benzenedisulfonimide (**1**, 1.10 g). The very exothermic reaction was stopped after stirring at r.t. for 5 min. The usual workup gave virtually pure octyl acetate (GC, GC-MS, ^{1}H and ^{13}C NMR) (16.84 g; 98%) as a colorless oil and *o*-benzenedisulfonimide was recovered in 91% yield (1.00 g; virtually pure: ^{1}H and ^{13}C NMR).[57]

The acylation products **4a**–**e** and **4g**–**z** are known compounds. They were characterized by GC-MS, ^{1}H and ^{13}C NMR spectra and by comparison with those reported in literature (for **4e**, **4h**, **4i**, **4j**, **4l**, **4n**, **4s**, **4t**, **4u**, **4v**, and **4w**) or with those of available commercial samples (for **4a**, **4d**, **4g**, **4k**, **4m**, **4o**, **4p**, **4q**, and **4r**); spectral properties for **4b**–**c** and **4x**–**z** are not yet reported in the literature. Details for the reactions and yields of the pure (GC, GC-MS, TLC, ^{1}H NMR) isolated products are listed in Table 1.

1-Naphthyl Acetate (4a)[3]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.41 (s, 3 H), 7.20 (d, *J* = 7.6 Hz, 1 H), 7.38–7.49 (m, 3 H), 7.70 (d, *J* = 8.4 Hz, 1 H), 7.80–7.85 (m, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.24, 118.30, 121.33, 125.62, 126.27, 126.67 (2 C), 126.97, 128.276, 134.85, 146.79, 169.78.

MS (EI, 70 eV): *m*/*z* = 186 (25) [M*], 144 (100).

1-Naphthyl Propanoate (4b)[58]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.34 (t, *J* = 7.6 Hz, 3 H), 2.73 (q, *J* = 7.6 Hz, 2 H), 7.22 (d, *J* = 7.4 Hz, 1 H), 7.38–7.50 (m, 3 H), 7.70 (d, *J* = 8.2 Hz, 1 H), 7.81–7.86 (m, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 9.52, 28.00, 118.28, 121.35, 125.64, 126.12, 126.62 (2 C), 127.11, 128.26, 134.84, 146.89, 173.14.

MS (EI, 70 eV): *m*/*z* = 200 (20) [M$^+$], 144 (100).

1-Naphthyl Benzoate (4c)[59]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.29–7.88 (m, 10 H), 7.90–8.35 (m, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 118.45, 121.48, 125.69, 126.29, 126.69 (2 C), 127.22, 128.27, 128.94 (2 C), 129.62, 130.53 (2 C), 133.98, 134.90, 147.06, 165.39.

MS (EI, 70 eV): *m*/*z* = 248 (15) [M$^+$], 115 (20), 105 (100).

Phenyl Acetate (4d)[60]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.24 (s, 3 H), 7.03 and 7.04 (2 d overlapped, *J* = 8.6, 8.2 Hz, 2 H), 7.17 (t, *J* = 7.6 Hz, 1 H), 7.33 (t, *J* = 7.2 Hz, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.35, 121.78 (2 C), 126.04, 129.64 (2 C), 152.20, 169.73.

MS (EI, 70 eV): *m*/*z* = 136 (20) [M$^+$], 94 (100).

***S*-Phenyl Thioacetate (4e)[3]**

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.36 (s, 3 H), 7.30–8.45 (m, 5 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 30.43, 128.14, 129.43 (2 C), 129.66, 134.68 (2C), 194.28.

MS (EI, 70 eV): *m*/*z* = 152 (30) [M$^+$], 110 (100).

2,6-Dichlorophenyl Acetate (4f)

Colorless oil; bp 111–112 °C/2.4 mbar.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.33 (s, 3 H), 7.06 (dd, *J* = 8.6, 7.4 Hz, 1 H), 7.28 (d, *J* = 7.8 Hz, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 20.41, 127.37, 128.82 (2 C), 129.10 (2 C), 144.23, 167.51.

MS (EI, 70 eV): *m*/*z* = 204 (20) [M$^+$], 162 (100).

Octyl Acetate (4g)[61]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.81 (t, *J* = 5.8 Hz, 3 H), 1.15–1.35 (m, 10 H), 1.50–1.59 (m, 2 H), 1.97 (s, 3 H), 3.98 (t, *J* = 6.7 Hz, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 14.23, 21.17, 22.80, 26.09, 28.78, 29.38 (2 C), 31.95, 64.83, 171.39.

MS (EI, 70 eV): *m*/*z* = 173 (5) [M$^+$ + 1], 57 (100).

Octyl Benzoate (4h)[62]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.82 (t, *J* = 6.7 Hz, 3 H), 1.20–1.38 (m, 10 H), 1.63–1.78 (m, 2 H), 4.25 (t, *J* = 6.7 Hz, 2 H), 7.32–7.55 (m, 3 H), 7.96–8.03 (m, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 14.30, 22.85, 26.25, 28.92, 29.46 (2 C), 32.00, 65.34, 128.50 (2 C), 129.72 (2 C), 130.73, 132.97, 166.88.

MS (EI, 70 eV): *m*/*z* = 234 (5) [M$^+$], 123 (100).

***S*-Octyl Thioacetate (4i)[63]**

^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.76–0.83 (m, 3 H), 1.15–1.35 (m, 8 H), 1.40–1.62 (m, 4 H), 2.24 (s, 3 H), 2.78 (t, *J* = 7.0 Hz, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 14.27, 22.82, 29.02, 29.33 (3 C), 29.69, 30.81, 31.97, 196.23.

MS (EI, 70 eV): *m*/*z* = 188 (15) [M$^+$], 145 (100).

S-Octyl Thiobenzoate (4j)[64]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.82 (t, *J* = 6.7 Hz, 3 H), 1.20–1.35 (m, 10 H), 1.52–1.68 (m, 2 H), 3.01 (t, *J* = 7.2 Hz, 2 H), 7.32–7.52 (m, 3 H), 7.88–7.94 (m, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 14.28, 22.84, 29.36 (4 C), 29.77, 31.99, 127.36 (2 C), 128.72 (2 C), 133.45, 137.50, 192.26.

MS (EI, 70 eV): *m*/*z* = 250 (5) [M$^+$], 105 (100).

Benzyl Acetate (4k)[3]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.04 (s, 3 H), 5.05 (s, 2 H), 7.25–7.35 (m, 5 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.19, 66.50, 128.44 (3 C), 128.75 (2 C), 136.14, 171.06.

MS (EI, 70 eV): *m*/*z* = 150 (50) [M$^+$], 108 (100).

Benzyl Propanoate (4l)[65]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.10 (t, *J* = 7.6 Hz, 3 H), 2.33 (q, *J* = 7.6 Hz, 2 H), 5.07 (s, 2 H), 7.30 (m, 5 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 9.31, 27.80, 66.33, 128.38 (2 C), 128.75 (2 C), 136.32, 174.50.

MS (EI, 70 eV): *m*/*z* = 164 (45) [M$^+$], 91 (100).

Benzyl Benzoate (4m)[66]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 5.32 (s, 2 H), 7.31–7.69 (m, 8 H), 8.02–8.06 (m, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 66.89, 128.37, 128.44, 128.58, 128.81, 129.92, 130.37, 133.23, 136.29, 166.63.

MS (EI, 70 eV): *m*/*z* = 212 (45) [M$^+$], 105 (100).

S-Benzyl Thioacetate (4n)[3]

^{1}H NMR (200 MHz; $CDCl_3$): δ = 2.29 (s, 3 H), 4.06 (s, 2 H), 7.20–7.25 (m, 5 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 30.54, 33.65, 127.49, 128.85 (2 C), 129.02 (2 C), 137.81, 195.34.

MS (EI, 70 eV): *m*/*z* = 166 (35) [M$^+$], 91 (100).

3-Phenylprop-2-enyl Acetate (4o)[3]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.04 (s, 3 H), 4.67 (d, *J* = 6.4 Hz, 2 H), 6.24 (dd, *J* = 16.0, 6.4 Hz, 1 H), 6.60 (d, *J* = 16.0 Hz, 1 H), 7.19–7.35 (m, 5 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.22, 65.29, 123.35, 126.81 (2 C), 128.28, 128.81 (2 C), 134.41, 136.38, 171.05.

MS (EI, 70 eV): *m*/*z* = 176 (45) [M$^+$], 115 (100).

Geranyl Acetate (4p)[3]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.53 (s, 3 H), 1.61 and 1.63 (2 s overlapped, 6 H), 1.98–2.08 (m, 7 H), 4.518 (d, *J* = 7.04 Hz, 2 H), 4.98–5.07 (m, 1 H), 5.27 (t, *J* = 6.8 Hz, 1 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 16.45, 17.86, 21.24, 25.86, 26.46, 39.71, 61.58, 118.40, 123.91, 132.01, 142.46, 171.32.

MS (EI, 70 eV): *m*/*z* = 196 (2) [M$^+$], 69 (100).

Neryl Acetate (4q)[3]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.53 (s, 3 H), 1.60 (s, 3 H), 1.69 (s, 3 H), 1.93–2.07 (m, 7 H), 4.48 (d, *J* = 7.4 Hz, 2 H), 4.95–5.07 (m, 1 H), 5.28 (t, *J* = 7.3 Hz, 1 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 17.82, 21.24, 23.69, 25.86, 26.81, 32.33, 61.29, 119.28, 123.73, 132.35, 142.83, 171.28.

MS (EI, 70 eV): *m*/*z* = 196 (2) [M$^+$], 69 (100).

(–)-Menthyl Acetate (4r)[3]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.66 (d, *J* = 6.8 Hz, 3 H), 0.79 (d, *J* = 7.2 Hz, 6 H), 0.80–1.05 (m, 3 H), 1.18–1.40 (m, 2 H), 1.50–1.65 (m, 2 H), 1.72–1.90 (m, 2 H), 1.93 (s, 3 H), 4.57 (dt, *J* = 10.8, 4.4 Hz, 1 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 16.51, 20.89, 21.45, 22.17, 23.62, 26.44, 31.52, 34.41, 41.08, 47.14, 74.26, 170.80.

MS (EI, 70 eV): *m*/*z* = 199 (2) [M$^+$ + 1], 95 (100).

Oct-1-en-3-yl Acetate (4s)[67]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.81 (t, *J* = 6.6 Hz, 3 H), 1.16–1.26 (m, 6 H), 1.50–1.60 (m, 2 H), 1.99 (s, 3 H), 5.06–5.22 (m, 3 H), 5.71 (ddd, *J* = 17.0, 10.4, 6.3 Hz, 1 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 14.15, 21.42, 22.68, 24.89, 31.72, 34.32, 75.04, 116.65, 136.85, 170.55.

MS (EI, 70 eV): *m*/*z* = 155 (2) [M$^+$ – 15], 99 (100).

Cyclohex-2-enyl Acetate (4t)[68]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.47–1.84 (m, 6 H), 1.93 (s, 3 H), 5.10–5.15 (m, 1 H), 5.54–5.62 (m, 1 H), 5.79–5.88 (m, 1 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 18.98, 21.51, 24.99, 28.41, 68.19, 125.83, 132.77, 170.86.

MS (EI, 70 eV): *m*/*z* = 140 (10) [M$^+$], 79 (100).

1-Phenylethyl Acetate (4u)[3,31]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.48 (d, *J* = 6.6 Hz, 3 H), 2.02 (s, 3 H), 5.83 (q, *J* = 6.6 Hz, 1 H), 7.25–7.31 (m, 5 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.57, 22.41, 72.51, 126.29 (2 C), 128.07, 128.69 (2 C), 141.87, 170.54.

MS (EI, 70 eV): *m*/*z* = 164 (40) [M$^+$], 122 (100).

1-Phenylethyl Propanoate (4v)[69]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7.4 Hz, 3 H), 1.48 (d, *J* = 6.6 Hz, 3 H), 2.30 (q, *J* = 7.4 Hz, 2 H), 5.85 (q, *J* = 6.6 Hz, 1 H), 7.22–5.35 (m, 5 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 9.30, 22.50, 28.08, 72.28, 126.25 (2 C), 128.01, 128.69 (2 C), 142.07, 173.90.

MS (EI, 70 eV): *m*/*z* = 178 (35) [M$^+$], 105 (100).

Diphenylmethyl Acetate (4w)[31]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.12 (s, 3 H), 6.86 (s, 1 H), 7.23–7.32 (m, 10 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.52, 77.09, 127.32 (4 C), 128.14 (2 C), 128.74 (4 C), 140.43 (2 C), 170.25.

MS (EI, 70 eV): *m*/*z* = 226 (25) [M$^+$], 165 (100).

2-Methyl-1-phenylpropan-2-yl Acetate (4x)[3]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.38 (s, 6 H), 1.91 (s, 3 H), 2.99 (s, 2 H), 7.10–7.23 (m, 5 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 22.76, 26.17 (2 C), 46.62, 82.26, 126.61, 128.13 (2 C), 130.73 (2 C), 137.53, 170.84.

MS (EI, 70 eV): *m*/*z* = 177 (5) [M$^+$ – 15], 132 (94), 91 (100).

1,1,1-Trichloro-2-methylpropan-2-yl Acetate (4y)[70]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.84 (s, 6 H), 2.04 (s, 3 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.38 (2 C), 22.40, 88.85, 106.10, 169.30.

MS (EI, 70 eV): *m*/*z* = 203 (10) [M$^+$ – 15], 123 (80), 59 (100).

1,1,1-Trichloro-2-methylpropan-2-yl Benzoate (4z)[71]

^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.99 (s, 6 H), 7.39 (t, *J* = 7.3 Hz, 2 H), 7.50 (app q, *J* = 7.2 Hz, 1 H), 8.00 (d, *J* = 7.5 Hz, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 21.61 (2 C), 89.39, 106.65, 128.66 (2 C), 130.03 (2 C), 130.86, 133.41, 164.62.

MS (EI, 70 eV): *m*/*z* = 280 (5) [M$^+$], 105 (100).

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