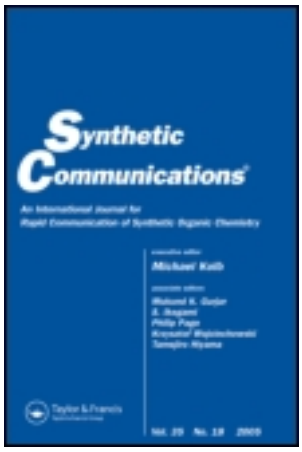

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SILICA GEL SUPPORTED JONES REAGENT (SJR): A SIMPLE, VERSATILE, AND EFFICIENT REAGENT FOR OXIDATION OF ALCOHOLS IN NON-AQUEOUS MEDIA

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SILICA GEL SUPPORTED JONES REAGENT (SJR): A SIMPLE, VERSATILE, AND EFFICIENT REAGENT FOR OXIDATION OF ALCOHOLS IN NON-AQUEOUS MEDIA

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ABSTRACT

Oxidation of alcohols to carbonyl compounds has been carried out by utilization of Jones reagent supported on silica gel (**SJR**). The **SJR** procedure has advantages over traditional Jones oxidation procedure. This procedure is safer to carry out and simplified workup by allowing organic media.

Chromium (VI) compounds are very effective in oxidizing alcohols to the corresponding carbonyl compounds. Among various Cr (VI) based oxidizing agents, Jones reagent is powerful and is capable of oxidizing almost any oxidizable organic compound or functional group.[1] Jones reagent has

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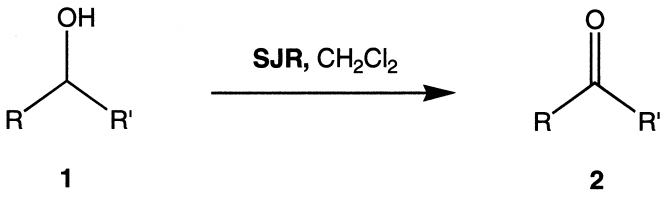
not been widely used in organic synthesis due to the toxicity associated with the reagent and its by-product. Some of the disadvantages associated with using Jones reagent are the potential health hazards from handling this reagent, problems in disposal of the toxic chromium compounds, and difficulty in product isolation. Recently, interest has been shown in utilization of solid-supported chromium reagents in oxidation reactions.[2] Solid supported chromium reagents have shown promise in circumventing many of the problems associated with the conventional reaction conditions for Jones reagent which utilize acetone–water as the reaction media. Solid supported chromium reagents offer advantages over the conventional methods in areas such as efficiency, selectivity and product isolation. Two methods have been reported for preparation of solid-supported chromium reagents: co-grinding CrO_3 with a solid support[3] and deposition of CrO_3 on a solid support by removal of the solvent from a mixture of the solid support and a solution of the chromium reagent.[4–12] These methods suffer from a number of disadvantages: cumbersome preparation,[7–10] light and moisture sensitivity,[4–6,10] loss of activity upon storing,[3,4,6] the necessity of activation by heating at a high temperature for prolonged period of time,[3–6,8] slow reaction,[5,10,12] and poor yields.[3–5,8] Also, the possibility of coming into contact with the toxic chromium reagent during preparation and use has hindered wide acceptance of these chromium-based oxidizing reagents. Thus, an efficient chromium reagent that is simpler and safer to prepare and use will benefit organic chemists.

In the course of our investigation of reactions on solid support,[13–17] we have developed a novel-oxidizing reagent: silica gel supported Jones Reagent (**SJR**). This reagent has been found to affect oxidation of a number of functional groups, including alcohol.[17]



1 **2**

SJR = Silica Gel Supported Jones Reagent

Preparation of **SJR** is very simple: a measured amount of Jones reagent and flash chromatography grade silica gel are mixed in a flash. It

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takes only a few minutes to prepare the reagent and does not require activation prior to using. **SJR** is much safer to prepare and use than existing chromium reagents. **SJR** can be prepared just before the reaction is carried out so there is no need for storing the reagent. However, in contrast to the earlier reports that chromic acid adsorbed on a solid support remains active only for one to two weeks if stored under vacuum in the dark,[4,6] we found that the **SJR** remains active over several months under ambient conditions.[18] Another big advantage of our procedure is that the amount of Jones reagent present on the silica gel may be controlled precisely. For other supported chromium reagents reported in the literature, the amount of chromium reagent loaded on the solid support has to be determined by titration. According to the procedure reported in this paper, a heterogeneous mixture of **SJR** and alcohol in methylene chloride solvent is stirred to achieve oxidation. (See the experimental for detail procedure). After the reaction is complete, the reaction mixture is filtered to remove silica gel along with the chromium compounds absorbed on it. The solid residue is washed with 60–75 mL of methylene chloride. Methylene chloride is not the only solvent that can be used in this reaction. We have found that the oxidation reactions work well in a non-polar solvent such as hexane. However, if hexane is used as the reaction media a polar solvent such as ethyl acetate or methylene chloride should be used to wash the solid residue in order to obtain good yields. The products obtained after the solvent is removed are often pure by NMR and TLC and do not require further purification. However, impure products were purified by radial chromatography using a mixture of ethyl acetate and hexane as the eluent. Oxidation reaction of alcohols with the **SJR** reported here are very fast, almost instantaneous at room temperature, and produces excellent yield of carbonyl product. **SJR** is very effective in oxidizing a wide variety of secondary alcohols with various degree of complexities to the corresponding ketones and primary alcohols to aldehydes. Alcohol functional groups are oxidized selectively to the corresponding ketones in the presence of carbon-carbon multiple bonds **2g**. Oxidation of benzoin **1m** and 1,2-diphenyl-1,2-ethanediol **1q** produced benzaldehyde in significant amount or as the only product. These results are similar to the report on oxidation of α-hydroxy carbonyl and 1,2-diols reported in the literature.[19,20] Our attempts to oxidize primary alcohols to aldehydes were met with limited success. Primary alcohols containing more than six carbons (**1s–1u**) produced good yield of aldehydes. Oxidation of alcohols containing less than six carbons was not clean and failed to produce acceptable yields of carbonyl compounds. The filtrate collected, after the reaction was complete, was orange in color and indicated the presence of chromium reagent in it. We believe, solubility of Jones reagent in low molecular weight alcohols and

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Table. Oxidation of Secondary Alcohols to the Ketones

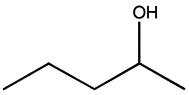
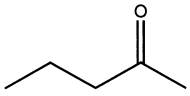
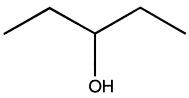
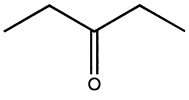
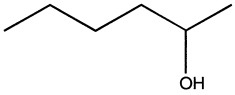
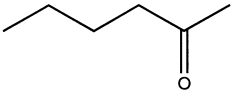
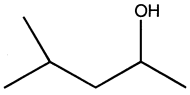
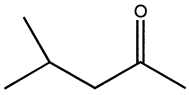
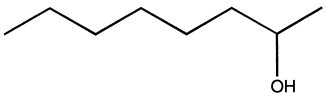
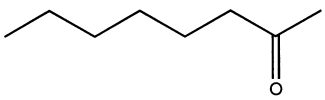
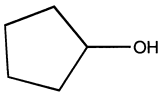
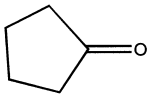
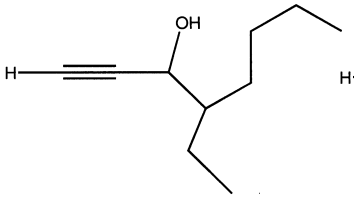
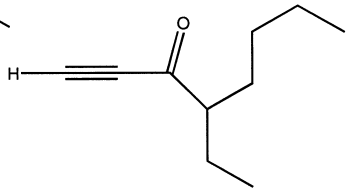
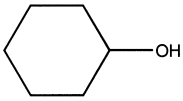
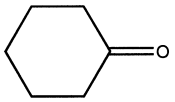
	Alcohol (**1**)	Ketone (**2**)	Yield (%)
a.			93
b.			80
c.			98
d.			78
e.			93
f.			94
g.			71
h.			96

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Table. Continued

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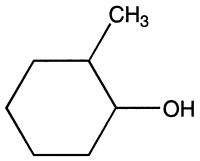
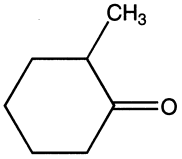
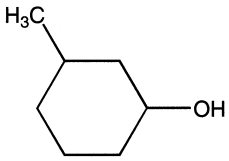
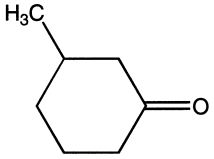
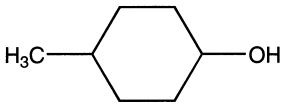
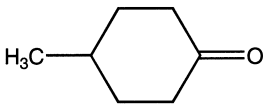
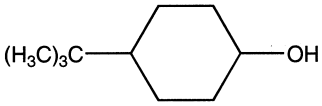
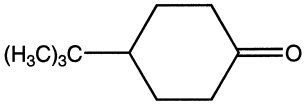
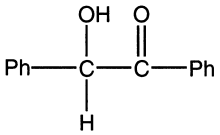
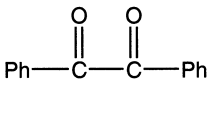
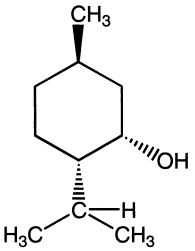
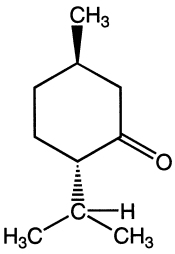
i.			99
j.			97
k.			98
l.			100
m.			72*
n.			100

(*continued*)

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Table. Continued

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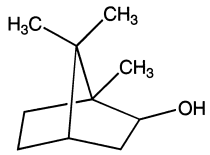
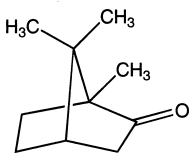
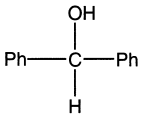
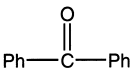
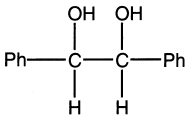
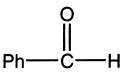
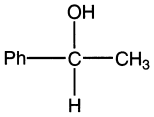
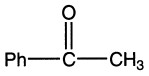
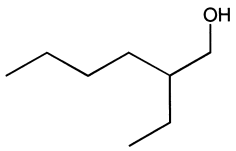
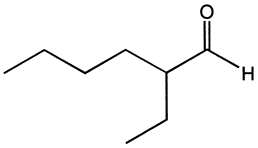
o.			89
p.			99
q.			72
r.			94
s.	$CH_3(CH_2)_6CH_2OH$	$CH_3(CH_2)_6CHO$	94
t.	$CH_3(CH_2)_8CH_2OH$	$CH_3(CH_2)_8CHO$	86
u.			76

* In addition to the 72% benzil, 13% benzaldehyde was isolated.

in carbonyl compounds complicated the reaction and also is responsible for the lower yields. Oxidation of low molecular weight secondary alcohols (C_2–C_4) also suffered from similar drawback. The bulkiness of the alcohol does not affect the efficacy of this reagent. The use of **SJR** eliminates or minimizes a number of problems associated with the classical Jones oxidation reaction including formation of an insoluble semi-solid chromium

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(III) salt by-product. We have found that **SJR** is superior to the previously reported chromium reagents on solid supports in terms of ease of preparation, selectivity and efficiency. Utilization of **SJR** in the oxidation of alcohols has made product isolation and disposal of waste easier. Very little handling of the chromium reagent is involved during the preparation of **SJR**. Also, aqueous work-up and extraction of product from the aqueous media are eliminated. Therefore concerns for users coming in contact with the toxic chromium reagents are minimized. In this procedure, a small amount of easy to dispose, solid waste is generated. Therefore, this newly developed silica gel supported Jones reagent is safer to use and generate lesser amounts of toxic waste.

EXPERIMENTAL

All reaction mixtures were stirred using Teflon coated magnetic stirring bars. Methylene chloride was used as received from the supplier without any further purification. Alcohols utilized in this study were purchased from Aldrich, Lancaster and Acros Chemical Company. The alcohols were used as they were received without further purification. All products were known compounds and were identified by comparison of the NMR and IR data with those reported in the literature or of authentic commercial products. Jones reagent was prepared according to the procedure in ref. 1. The silica gel, used as solid support, was MN-Kieselgel 60 (0.04–0.063 mm mesh size) supplied by Fisher Scientific. Proton NMR spectra were recorded on a Bruker DPX-300 NMR instrument. Samples for NMR were dissolved in $CDCl_3$. Proton chemical shifts are expressed as ppm relative to tetramethylsilane. IR spectra were recorded on a Perkin–Elmer Spectrum 1000 FT-IR instrument and are reported in wave numbers (cm^{-1}). Chromatographic separations were carried out by preparative centrifugal thin-layer chromatography with silica gel (Merck #7749) on a Chromatotron Model 7924T. Analytical thin-layer chromatography was done on pre-coated silica gel plates with 254 nm fluorescent indicator (Merck #5715) and developed in ethyl acetatehexanes (1 : 4) mixed solvent system. Compounds were visualized by a UV lamp and/or by staining either with *p*-anisaldehyde/sulfuric acid or phosphomolybdic acid.

General Procedure

A 100 mL round bottom flask fitted with a rubber septum was charged with 5 gm flash chromatographic grade silica gel. 1.5 mL of Jones reagent

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(12 mmols) was added drop-by-drop using a syringe while the content of the flask was stirred using a magnetic stirring bar. After the addition of the Jones reagent was complete, stirring of the silica gel continued until a free flowing orange solid was obtained, which took less than 5 min. 25 mL of methylene chloride was then added to the flask and the heterogeneous mixture was stirred. A solution of the alcohol (3–4 mmoles) in about 2 mL of methylene chloride was then added to the reaction mixture. The orange color of the supported reagent turned dark immediately upon addition of the alcohol solution. After stirring for 5 min the reaction mixture was filtered through a sintered-glass funnel. The solid was washed with about 75 mL of methylene chloride. Removing the solvent from the combined methylene chloride solutions isolated products. The isolated products were often pure by proton NMR and TLC. Impure products were purified by employing radial chromatography using a 1 : 9 mixture of ethyl acetate and hexane as the eluent.

2-Pentanone (**2a**)[21] ^{1}H NMR (300 MHz, $CDCl_3$) 0.94 (t, $j = 6.5$ Hz, 3H), 1.60 (m, 2H), 2.13 (s, 3H), 2.41 (t, $J = 7.20$ Hz, 2H); ^{13}C NMR 13.44, 17.06, 29.61, 45.42, 108.92; IR (neat) 734, 918, 963, 1170, 1237, 1272, 1365, 1410, 1459, 1715, 2877, 2937, 2965 cm^{-1}.

3-Pentanone (**2b**)[21] ^{1}H NMR (300 MHz, $CDCl_3$) 1.05 (t, $j = 7.4$ Hz, 6H), 2.43 (q, $j = 7.4$ Hz, 4H); ^{13}C NMR 7.60, 35.20, 211.90; IR (neat) 734, 812, 918, 956, 1094, 1122, 1171, 1358, 1377, 1415, 1460, 1716, 2884, 2908, 2940, 2979 cm^{-1}.

2-Hexanone (**2c**)[21] ^{1}H NMR (300 MHz, $CDCl_3$) 0.90 (t, $j = 7.2$ Hz, 3H), 1.33 (m, 2H), 1.57 (m, 2H), 2.15 (s, 3H), 2.44 (t, $j = 7.5$ Hz, 2H); ^{13}C NMR 13.45, 22.00, 25.62, 29.43, 43.17, 209.89; IR (neat) 590, 801, 881, 1050, 1089, 1170, 1261, 1368, 1410, 1456, 1712, 2876, 2934, 2967 cm^{-1}.

4-Methyl-2-pentanone (**2d**)[21] ^{1}H NMR (300 MHz, $CDCl_3$) 0.93 (d, $j = 6.7$ Hz, 6H), 2.12 (s, 3H), 2.05–2.17 (m, 1H), 2.31 (d, $j = 6.95$, 2H); ^{13}C NMR 22.61 (2C), 24.67, 30.36, 52.79, 208.6; IR (neat) 649, 732, 911, 1170, 1242, 1367, 1467, 1710, 2873, 2935, 2961 cm^{-1}.

2-Octanone (**2e**)[21] ^{1}H NMR (300 MHz, $CDCl_3$) 0.88 (t, $j = 6.6$ Hz, 3H), 1.30 (m, 6H), 1.58 (m, 2H), 2.14 (s, 3H), 2.43 (t, $j = 7.4$ Hz, 2H); ^{13}C NMR 14.14, 22.60, 23.90, 28.96, 29.81, 31.73, 43.80, 209.13; IR (neat) 719, 946, 1035, 1114, 1165, 1225, 1277, 1361, 1411, 1466, 1717, 2858, 2930, 2957 cm^{-1}.

Cyclopentanone (**2f**)[21] ^{1}H NMR (300 MHz, $CDCl_3$) 1.97 (m, 4H), 2.15 (m, 4H); ^{13}C NMR 23.28, 38.36, 220.54; IR (neat) 741, 833, 913, 959, 1047, 1152, 1231, 1267, 1312, 1407, 1454, 1746, 2884, 2966 cm^{-1}.

4-Ethyl-1-octyn-3-one (**2g**)[21] ^{1}H NMR (300 MHz, $CDCl_3$) 0.89 (t, $j = 7.1$ Hz, 3H), 0.90 (t, $j = 7.4$ Hz, 3H), 1.20–1.35 (m, 4H), 1.40–1.80 (m, 4H), 2.43 (m, 1H), 3.26 (s, 1H); ^{13}C NMR 11.57, 13.89, 22.70, 24.26, 29.33, 30.50, 56.04, 78.93, 80.67, 191.68; IR (neat) 741, 813, 896, 1020, 1072,

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1188, 1266, 1382, 1422, 1460, 1676, 1719, 2093, 2861, 2874, 2934, 2963, 3293 cm^{-1}.

Cyclohexanone (**2h**)[21] ^1H NMR (300 MHz, $CDCl_3$) 1.74 (m, 2H), 1.86 (m, 4H), 2.35 (m, 4H); ^{13}C NMR 25.03, 27.09 (2C), 41.99 (2C), 211.94; IR (neat) 652, 749, 864, 908, 1017, 1118, 1221, 1311, 1338, 1422, 1449, 1714, 2863, 2938 cm^{-1}.

2-Methylcyclohexanone (**2i**)[21] ^1H NMR (300 MHz, $CDCl_3$) 1.03 (d, $j = 6.6$ Hz, 3H), 1.36 (m, 1H), 1.64 (m, 2H), 1.83 (m, 1H), 2.07 (m, 2H), 2.30–2.43 (m, 3H); ^{13}C NMR 14.74, 25.22, 28.01, 36.23, 41.88, 45.38, 213.55; IR (neat) 647, 732, 816, 865, 918, 990, 1052, 1155, 1215, 1313, 1376, 1428, 1449, 1722, 2861, 2927 cm^{-1}.

3-Methylcyclohexanone (**2j**)[21] ^1H NMR (300 MHz, $CDCl_3$) 1.02 (d, $j = 6.2$ Hz, 3H), 1.37 (m, 1H), 1.64–1.70 (m, 1H), 1.84–2.10 (m, 4H), 2.20–2.41 (m, 3H); ^{13}C NMR 22.06, 25.32, 33.28, 34.21, 41.09, 49.96, 211.62; IR (neat) 751, 817, 866, 960, 1055, 1116, 1224, 1275, 1317, 1344, 1360, 1421, 1456, 1715, 2870, 2928, 2955 cm^{-1}.

4-Methylcyclohexanone (**2k**)[21] 1.02 (d, $j = 6.20$ Hz, 3H), 1.36–1.50 (m, 2H), 1.82–2.10 (m, 3H), 2.29–2.40 (m, 4H); ^{13}C NMR 20.42, 30.53, 34.22, 40.20, 211.03; IR (neat) 745, 908, 957, 1003, 1122, 1184, 1248, 1331, 1421, 1459, 1717, 2870, 2929, 2955 cm^{-1}.

4-*tert*-Butylcyclohexanone (**2l**)[21] ^1H NMR (300 MHz, $CDCl_3$) 0.91 (s, 9H), 1.39–1.51 (m, 4H), 2.03–2.12 (m, 1H), 2.26–2.40 (m, 4H); ^{13}C NMR 27.61, 32.46, 41.30, 46.70, 212.46; IR (neat) 667, 703, 737, 841, 944, 1162, 1219, 1332, 1366, 1420, 1449, 1471, 1718, 2870, 2911, 2954 cm^{-1}.

Benzil (**2m**)[21] ^1H NMR (300 MHz, $CDCl_3$) 7.50 (m, 4H), 7.63 (m, 2H), 7.96 (m, 4H); ^{13}C NMR 128.87, 129.69, 132.76, 134.78, 194.23; IR (film) 683, 717, 795, 875, 998, 1071, 1172, 1210, 1323, 1450, 1579, 1594, 1658, 3029, 3064 cm^{-1}.

(d,l)-Menthone (**2n**)[21] ^1H NMR (300 MHz, $CDCl_3$) 0.86 (d, J = 6.7 Hz, 3H), 0.91 (d, J = 6.7 Hz, 3H), 1.01 (d, J = 6.2 Hz, 3H), 1.34–1.40 (m, 2H), 1.80–2.2 (m, 6H), 2.18–2.37 (m, 1H) ppm; ^{13}C NMR 18.72, 21.22, 22.31, 25.92, 27.90, 33.96, 35.50, 50.87, 55.83, 212.09 ppm; IR (film) 608, 747, 866, 994, 1044, 1093, 1117, 1155, 1246, 1286, 1367, 1457, 1711, 2871, 2928, 2956 cm^{-1}.

(d,l)-Camphor (**2o**)[21] ^1H NMR (300 MHz, $CDCl_3$) 0.84 (s, 3H), 0.91 (s, 3H), 0.96 (s, 3H), 1.13–1.47 (m, 2H), 1.60–1.73 (m, 1H), 1.85 (d, $j = 18.3$ Hz, 1H), 1.90–1.98 (m, 1H), 2.09 (t, $j = 4.5$ Hz, 1H), 2.30–2.40 (dt, $j = 3.84$, 18.3 Hz, 1H); ^{13}C NMR 9.26, 19.15, 19.78, 27.05, 29.90, 43.03, 43.29, 46.78, 57.69, 219.66; IR (film) 646, 748, 851, 944, 1020, 1044, 1092, 1276, 1322, 1372, 1390, 1416, 1447, 1742, 2873, 2960 cm^{-1}.

Benzophenone (**2p**)[22] ^1H NMR (300 MHz, $CDCl_3$) 7.44 (m, 2H), 7.51–7.57 (m, 1H), 7.78 (m, 2H); ^{13}C NMR 128.23, 129.98, 132.37, 137.52,

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196.60; IR (film) 638, 697, 763, 810, 919, 941, 999, 1028, 1074, 1150, 1176, 1276, 1317, 1447, 1577, 1598, 1658, 3060 cm^{-1}.

Benzaldehyde (**2q**)[22] ^1H NMR (300 MHz, $CDCl_3$) 7.40–7.46 (m, 2H), 7.50–7.63 (m, 1H), 7.80–7.88 (dt, $J = 6.83$ Hz, $J = 1.5$ Hz, 1H), 9.96 (s, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) 192.39, 136.46, 134.44, 129.68, 129.02 ppm. IR (film) 650, 688, 746, 828, 1023, 1167, 1204, 1311, 1391, 1455, 1584, 1597, 1654, 1702, 2738, 2820, 2852, 3031, 3064 cm^{-1}.

Acetophenone (**2r**)[22] ^1H NMR (300 MHz, $CDCl_3$) 2.55 (s, 3H), 7.38–7.46 (m, 2H), 7.48–7.55 (m, 1H), 7.90–7.95 (m, 2H) ppm; ^{13}C NMR 26.53, 128.27, 128.55, 133.08, 137.05, 198.02 ppm; IR (film) 690, 760, 955, 1024, 1078, 1180, 1266, 1359, 1449, 1582, 1599, 1686, 2924, 3005, 3063 cm^{-1}.

Octanal (**2s**)[21] ^1H NMR (300 MHz, $CDCl_3$) 0.88 (bs, 3H), 1.29 (bs, 8H), 1.62 (bs, 2H), 2.42 (t, $j = 8$ Hz, 1H), 2.43 (t, $j = 8$ Hz, 1H), 9.76 (t, $j = 8$ Hz, 1H) ppm; ^{13}C NMR 14.0, 22.1, 22.6, 29.05, 29.15, 31.66, 43.94, 203 ppm; IR (film) 723, 947, 1078, 1379, 1429, 1465, 1728, 2714, 2857, 2928, 2956 cm^{-1}.

Decanal (**2t**)[21] ^1H NMR (300 MHz, $CDCl_3$) 0.88 (t, $j = 7.6$ Hz, 3H), 1.15–1.35 (bs, 14H), 1.60 (bs, 2H), 2.40 (t, $j = 8$ Hz, 1H), 2.41 (t, $j = 8$ Hz, 1H), 9.86 (t, $j = 8$ Hz, 1H) ppm; ^{13}C NMR 14.2, 22.2, 22.7, 29.3, 29.38, 29.6, 29.71, 32.0, 44.1, 203 ppm; IR (film) 718, 890, 1058, 1369, 1412, 1468, 1722, 2721, 2851, 2976 cm^{-1}.

2-Ethylhexanal (**2u**)[21] ^{13}C NMR 0.91 (m, 6H), 1.16–1.80 (m, 8H), 2.21 (m, 1H), 9.76 (d, $j = 7.4$ Hz, 1H) ppm; ^{13}C NMR 11.5, 14.1, 21.92, 22.86, 28.3, 29.31, 53.45, 206.5 ppm; IR (film) 740, 821, 910, 1020, 1080, 1140, 1268, 1321, 1452, 1722, 2720, 2860, 2925, 2954 cm^{-1}.

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