Novel Brønsted Acidic Ionic Liquids and Their Use as Dual Solvent-Catalysts

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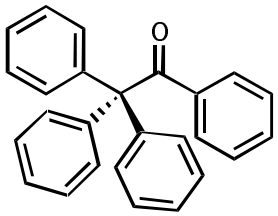
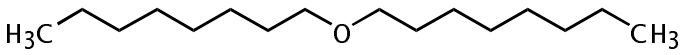
Supporting Information

General Considerations. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were obtained as solutions in either $CDCl_3$ or D_2O. Chemical shifts were reported in parts per million (ppm, δ) and referenced to $CHCl_3$ (δ 7.27) or D_2O (δ 4.88). Infrared spectra were recorded as a thin film on sodium chloride and absorptions were reported in wavenumbers (cm^{-1}). Melting points are uncorrected. Distillations were performed using a Kugelrohr ball-tube distillation apparatus. Gas chromatographic analyses were performed using an Agilent 6850 system (FID). TLC analyses were performed on Whatman flexible polyester backed TLC plates with a fluorescent indicator. Detection was conducted by UV absorption (254 nm) and charring with 10% $KMnO_4$ in water. Baker silica gel (47-61 microns) was used for all chromatographic separations. Anhydrous organic solvents were dried and then distilled prior to use. Acetic acid, acetic anhydride, benzopinacole, ethanol, hexanoic acid, 1-octanol, pinacol and *p*-toluenesulfonic acid were not purified prior to use. All other chemicals used for synthetic procedures were reagent grade or better. Solutions were concentrated in vacuo with a rotary evaporator and the residue was purified using a silica gel column unless specified otherwise.



Benzopinacolone (2,2,2-triphenylacetophenone) To a 5 mL reaction conical vial equipped with stir bar and reflux condenser was added 882 mg triphenyl(propyl-3-sulphonyl)phosphonium toluenesulfonate. Next added in one portion was 58.8 mg benzopinacole. The reaction was allowed to warm to 140•C for a period of 2 hours. The resulting monophase was then allowed to cool to room temperature at which time the biphase was washed with EtOAc (3 x 2.0 mL) after addition of 1.0 mL water and 2.0 mL EtOAc. The combined organic phases were dried with anhydrous $MgSO_4$, filtered and concentrated in vacuo. The crude product (71.1 mg) revealed over a 99% conversion from benzopinacole to benzopinacolone via GC ((HP-1 methyl siloxane) 100°C (2 min), 10°C/min, 275°C (10 min)) 11.32 min (benzopinacole); 21.38 min (benzopinacolone). Purification by silica gel chromatography (EtOAc/Hex 1:8) afforded the desired material in 49.5 mg (88% isolated yield) as a white crystalline solid. Spectral data of this material matched that of commercially available material.

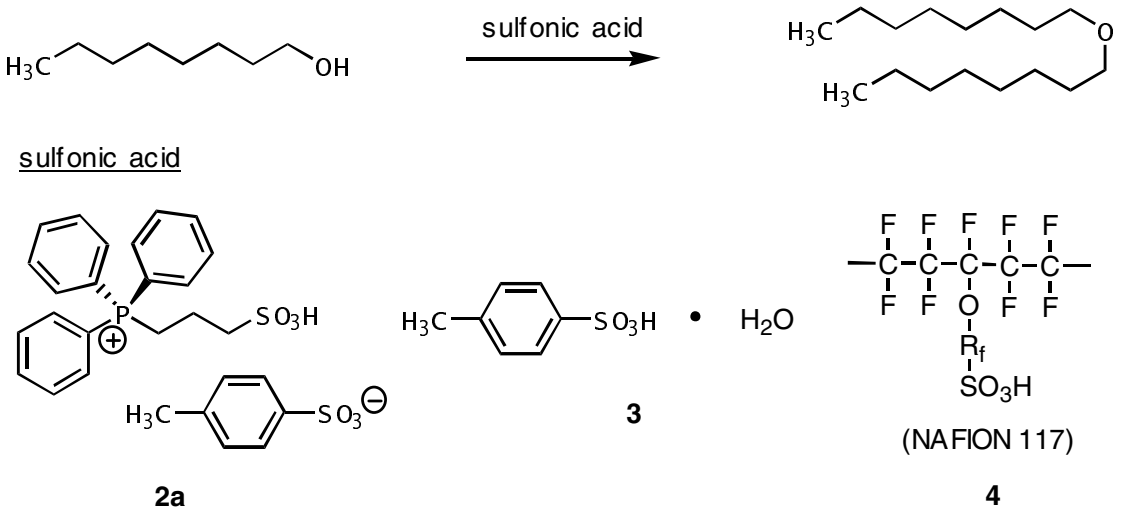


***n*-octyl ether** To a 5 mL reaction conical vial equipped with stir bar and reflux condenser was added 1.0 g (1.91 mmol) triphenyl(propyl-3-sulphonyl)phosphonium toluenesulfonate. Next added in one portion was 1.0 mL (6.35 mmol) 1-octanol. The reaction was allowed to warm to 175•C over a period of 2 hours. The resulting monophase was then allowed to cool to room temperature at which time the biphase was washed with EtOAc (3 x 2.0 mL) after addition of 1.0 mL water and 2.0 mL EtOAc. The combined organic phases were dried with anhydrous $MgSO_4$, filtered and concentrated in vacuo. Purification by bulb-to-bulb distillation (bp 130 °C/ 3 mm Hg (air bath temp)) afforded the desired material in 432 mg (56 % isolated yield) as a clear and colorless oil. The ratio of alcohol to IL did have a profound affect in overall yield of octyl ether formation. From the combination of approximately 300 mg (0.57 mmol) triphenyl(propyl-3-sulphonyl)phosphonium toluenesulfonate and 0.45 mL 1-octanol, afforded was 55 mg (16% isolated yield) whereas from the combination of 771 mg (1.47 mmol) triphenyl(propyl-3-sulphonyl)phosphonium toluenesulfonate and 0.5 mL (3.18 mmol) 1-octanol, afforded was 96.8 mg (25% isolated yield). The products in each run were analyzed by GC ((HP-1 methyl siloxane) 100°C (2 min), 10°C/min, 275°C (10 min)) 4.83min (1-octanol); 12.03 min (octyl ether) and confirmed by NMR. Spectral data of this material matched that of commercially available material.

Control Reaction using PTSA From the combination of 1-octanol (0.5 mL, 3.17 mmol) and *p*-toluenesulfonic acid (280 mg, 1.47 mmol (using the monohydrate)) was obtained 187 mg *n*-octyl ether (49% isolated yield) based upon purification of the crude product by bulb-to-bulb distillation (bp 130 °C/ 3 mm Hg (air bath temp)).

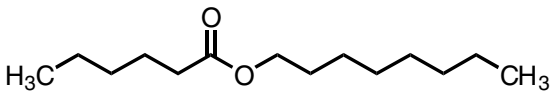
Control Reaction using NAFION 117 From the combination of 0.5 mL 1-octanol (3.17 mmol) and 0.314 g NAFION 117 (0.28 meq (0.89 meq/g)) in 3.0 mL toluene (1.1M) as solvent was obtained 12.4 mg *n*-octyl ether (3% isolated yield) upon purification of the crude product (GC ratio of 90:10 (octanol:octyl ether)) by bulb-to-bulb distillation (bp 130 °C/ 3 mm Hg (air bath temp)).

Table 1. Analysis of Octyl Ether Formation.

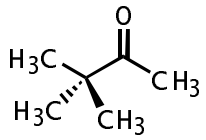


Entry	Sulfonic Acid Derivative	Ratio of 1-octanol to sulfonic acid derivative	Ratio of 1-octanol to octyl ether[a]	Isolated Yield, %
1	**2a**	2.2:1.0	54:46	25
2	**2a**	3.3:1.0	20:80	56
3	**2a**	5.0:1.0	77:23	16
4	**3**	2.2:1.0	15:85	49
5	**4**	-[b]	96:4	3

[a]Ratio based upon unreacted 1-octanol to isolated octyl ether (GC analysis of the distilled product). [b]0.28 meq which is based upon amount of 1-octanol (0.5 mL, 3.17 mmol) and NAFION (314 mg, 0.28 meq (0.89 meq/g)) used.

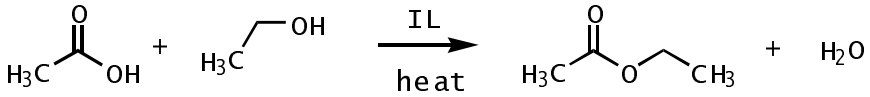


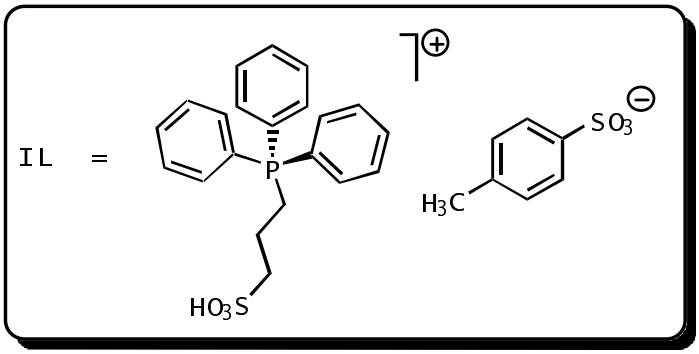
***n*-octyl hexanoate** To a 5 mL reaction vial equipped with stir bar was added approximately 200 μL of 3-butyl-1-(butyl-4-sulfonyl)imidazolium trifluoromethanesulfonate (1.9 M). Added next via syringe was 1-octanol (60 μL, 0.38 mmol) followed by hexanoic acid (48 μL, 0.38 mmol). The resulting monophase was allowed to stir at room temperature for a period of 7 days at which time the oil was washed with toluene (5 x 2 mL). Shorter reaction times using higher reaction temperatures also afforded excellent conversion of acid to ester. Reaction of 1-octanol and acetic acid resulted in 89% conversion to *n*-octyl acetate at 40°C for a period of 72h whereas 83% conversion was observed at 40°C for 48h. The collected organic washes were concentrated in vacuo to afford 76 mg of *n*-octyl hexanoate. GC analysis of the crude product revealed only trace amounts of starting material in the organic washes. Purification of the crude colorless oil by bulb-to-bulb distillation afforded the desired compound in 72 mg (0.31 mmol, 82% yield) as a clear and colorless oil (bp 130 °C/ 3 mm Hg (air bath temp)). ^{1}H NMR (300 MHz, $CDCl_3$); δ 4.04, J = 6.6, 2H), 2.27 (t, J = 7.4, 2H), 1.63 - 1.58 (m, 4H), 1.30 - 1.25 (m, 14H), 0.87 - 0.80 (m, 6H). ^{13}C NMR (75.5 MHz, $CDCl_3$); δ 174.10, 64.47, 34.42, 31.84, 31.39, 29.26, 28.72, 26.00, 24.78, 22.70, 22.38, 14.13, 13.96. IR (thin film) 2956, 2929, 2858, 1739, 1466, 1173 cm^{-1}. GC ((HP-1 methyl siloxane; f = 1.0 mL/min) 100°C (2 min), 10°C/min, 275°C (10 min)) 11.05 min.



Pinacolone (3,3-dimethyl-2-butanone) A 5 mL reaction conical vial was equipped with stir bar and Hinkman-Hinkle still head. Attached onto the still head was a reflux condenser with drying tube. To the reaction conical vial was added approximately 1.0 g triphenyl(propyl-3-sulphonyl)phosphonium toluenesulfonate. Next added in one portion to the reaction vial was 290 mg pinacol. The reaction was allowed to warm to a maximum temperature of 180•C for a total period of 1 hour. The resulting monophase was then allowed to cool to room temperature at which time the distillate was transferred to another flask and analyzed by GC ((HP-1 methyl siloxane) 50°C (2 min), 10°C/min, 275°C (10 min)) 4.07 min (pinacolone); 6.17 min (pinacol) and NMR. Purification of the crude product via bulb-to-bulb distillation (bp 125 °C/ 3 mm Hg (air bath temp)) afforded the desired material in 86 mg (35% isolated yield) as a clear and colorless oil. Spectral data of this material matched that of commercially available material.

Reuse of IL in the Formation of Ethyl Acetate





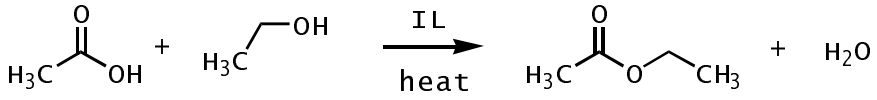
The reaction setup used to illustrate the reuse of the IL in synthetic transformations consisted of a 5 mL reaction conical vial equipped with a magnetic spin vane. Attached to the conical vial was a Hinkman-Hinkle still head which itself was equipped with a Claisen adapter and reflux condenser. Proper alignment of one of the two inlet ports of the Claisen adapter is essential for the addition of reagents via a syringe pump. The setup was equipped with a drying tube packed with $CaCl_2$ and heated externally via a sand bath.

Representative Procedure To the 5 mL reaction conical vial charged with 2.1 g IL (4.0 mmol) was added via syringe acetic acid (1.0 mL, 17.5 mmol) and ethanol (1.0 mL, 17.5 mmol). The reaction mixture was allowed to warm to a maximum temperature of 175°C (external temperature) over a period of 45 min. Although completion of reaction was observed prior to reaching the maximum temperature, reuse of the reaction setup/IL made it necessary to remove all volatile components via distillation prior to the next cycle. With each cycle, reaction completion was confirmed by GC analysis [GC ((HP-1 methyl siloxane; f = 1.0 mL/min) 50°C (2 min), 10°C/min, 275°C (10 min)) 3.21 min (EtOH), 3.37 min (EtOAc), 3.42 min (AcOH)] and documented via the mass of distillate obtained. Each cycle afforded pure ethyl acetate without any appreciable amounts of starting material (<7% assuming loss of EtOH due to an EtOH/EtOAc azeotrope (31% by weight and bp of 78°C)). Biphasic mixtures were separated and factored into product formation was maximum water content of 3.3%.

Control Reaction A 5 mL reaction conical vial was charged with acetic acid (1.0 mL, 17.5 mmol) and ethanol (1.0 mL, 17.5 mmol). The reaction mixture was allowed to warm to a maximum temperature of 175°C (external temperature) over a period of 45 min. Observed were no appreciable amounts of distillate even after continuous heating 30 min after the 45 min window. The distillate that was collected consisted of EtOH (97%) and EtOAc (7%). Remaining in the reaction conical vial was AcOH.

Regeneration and Subsequent Reuse of Ionic Liquid The system after multiple cycles retained a significant amount of mass, primarily water, which correlated to a rise in mass percentage of over 151%. Based upon the mass balance of reaction cycles and product yields, the rise in mass consists of water and acetic acid. Removal of the VOCs using heat (<175°C) at atmospheric pressure was unsuccessful. However, when the setup was evacuated (10 Torr) and warmed to 65°C over a period of 5 hours, observed was the loss in volume contained in the reaction vial. The resulting ionic liquid still contained AcOH (42%) based upon ^{1}H NMR analysis. Using the results from cycle 2, 0.441 mL of water was added prior to the addition of ethanol and acetic acid in an effort to mimic the water:IL ratio. This control experiment afforded 1.3 g ethyl acetate (87% isolated yield).

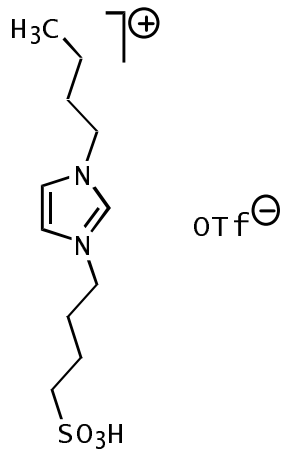
Table 2. Reuse of Ionic Liquid in the Formation of Ethyl Acetate.



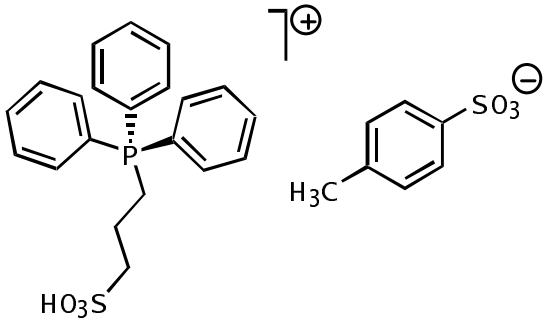
cycle	ethyl acetate, %[a]
1	82
2	91
3	96
4	81
5[b]	87

[a]Isolated yield. [b]Isolated yield using regenerated ionic liquid (addition of 0.441

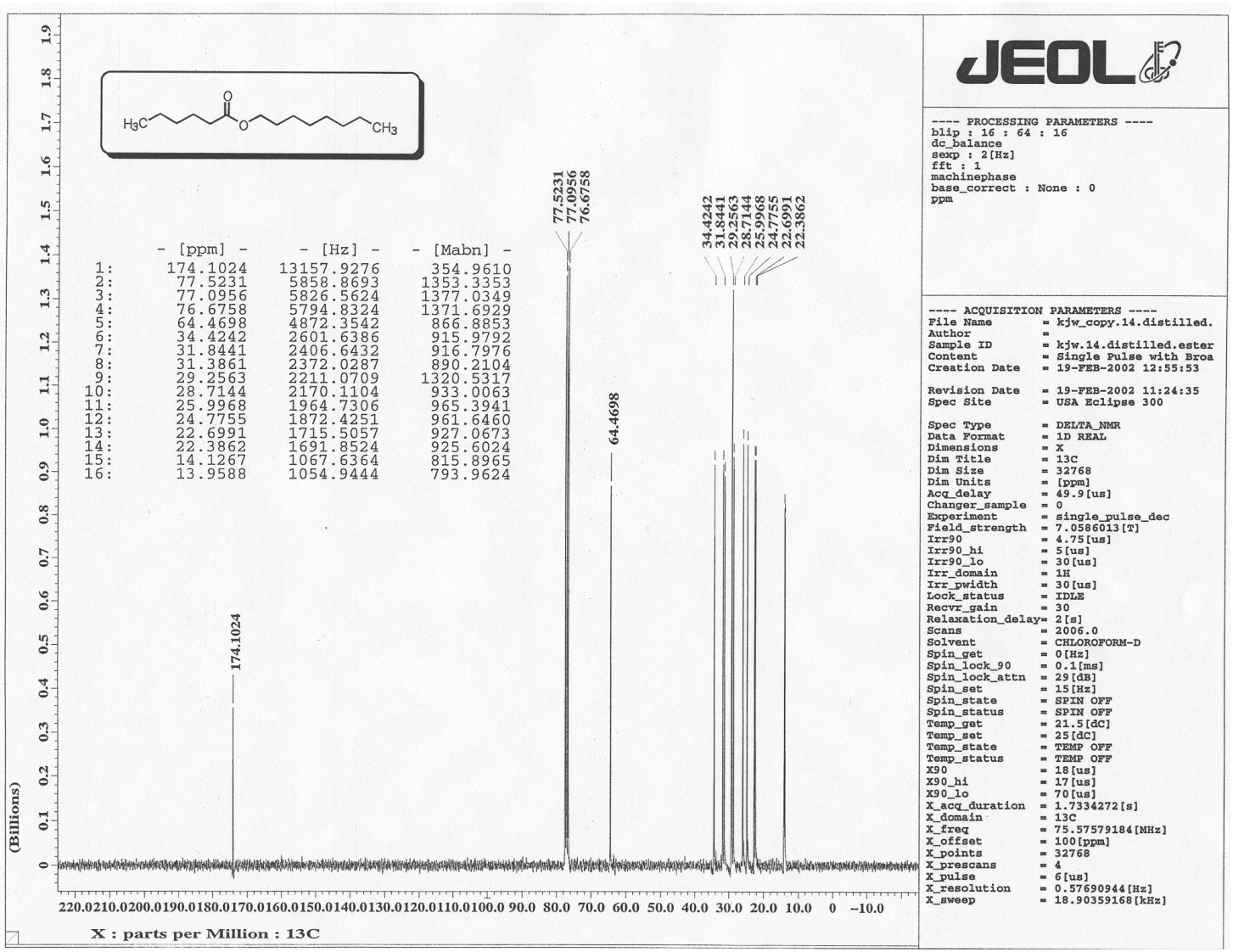
mL water prior to run).



3-butyl-1-(butyl-4-sulfonyl)imidazolium trifluoromethanesulfonate From the combination of 1-butylimidazole and 1,4-butane sultone was formed in excellent yield. After washing the salt with diethyl ether and toluene to remove any unreacted starting materials, the solid was dried in vacuo. Then, a stoichiometric amount of trifluoromethanesulfonic acid was added and the mixture stirred for two hours at 40°C during which time the solid zwitterion dissolved/liquefied, resulted in the formation of 3-butyl-1-(butyl-4-sulfonyl)imidazolium trifluoromethanesulfonate. The IL phase was then washed repeatedly with toluene and ether to remove non-ionic residues, and dried in vacuo. The product was formed quantitatively and in high purity as assessed by mass balance and NMR spectroscopy. Spectral data: ^{1}H NMR (300 MHz, D_2O); δ 8.68 (s, 1H), 7.40 (d, *J* = 1.6, 1H), 7.39 (d, *J* = 1.6, 1 H), 4.13 (t, *J* = 6.9, 2H), 4.08 (t, *J* = 7.1, 2H), 2.82 (t, *J* = 7.4, 2H), 1.91 (quint, *J* = 8.0, 2H), 1.73 (q, *J* = 7.7, 2H), 1.68 - 1.57 (m, 2H), 1.19 (dt, *J* = 7.7, 7.7, 2H), 0.79 (t, *J* = 7.4, 3H). ^{13}C NMR (75.5 MHz, D_2O) δ 135.26, 122.64, 122.42, 119.80 (q, $J_{C\text{-}F}$ = 317.0, CF_3), 50.22, 49.49, 49.10, 31.31, 28.26, 21.11, 18.88, 12.75.



triphenyl(propyl-3-sulphonyl)phosphonium toluenesulfonate Triphenylphosphine and 1,3-propane sultone are combined in equimolar quantities in toluene and brought to reflux. Overnight, a white precipitate forms which is isolated by filtration and dried. Analysis of the solid revealed it to be the desired zwitterion, formed in quantitative yield. The desired zwitterion was of sufficient purity to be used without any further purification. Convesrion to the ionic liquid is accomplished by combining equimolar quantities of pTSA hydrate and the zwitterion and heating to 70°C for 24h, during which time the solids liquefy, resulting in the formation of triphenyl(propyl-3-sulfonyl)phosphonium toluenesulfonate. The IL phase was then washed repeatedly with toluene and ether to remove non-ionic residues, and dried in vacuo. The product was formed quantitatively and in high purity as assessed by mass balance and NMR spectroscopy. Spectral data: ^{1}H NMR (300 MHz, D_2O); δ 7.66 - 7.60 (m, 3H), 7.53 - 7.44 (m, 14H), 7.06 (d, *J* = 8.0, 2H), 3.31 - 3.21 (m, 2H), 2.89 (t, *J* = 6.9, 2H), 2.11 (s, 3H), 1.97 - 1.80 (m, 2H). ^{13}C NMR (75.5 MHz, D_2O); δ 142.21, 139.75, 135.29, 135.25, 133.47, 133.34, 130.40, 130.23, 129.42, 125.44, 118.13, 116.98, 50.55, 50.33, 20.58, 20.04, 17.94.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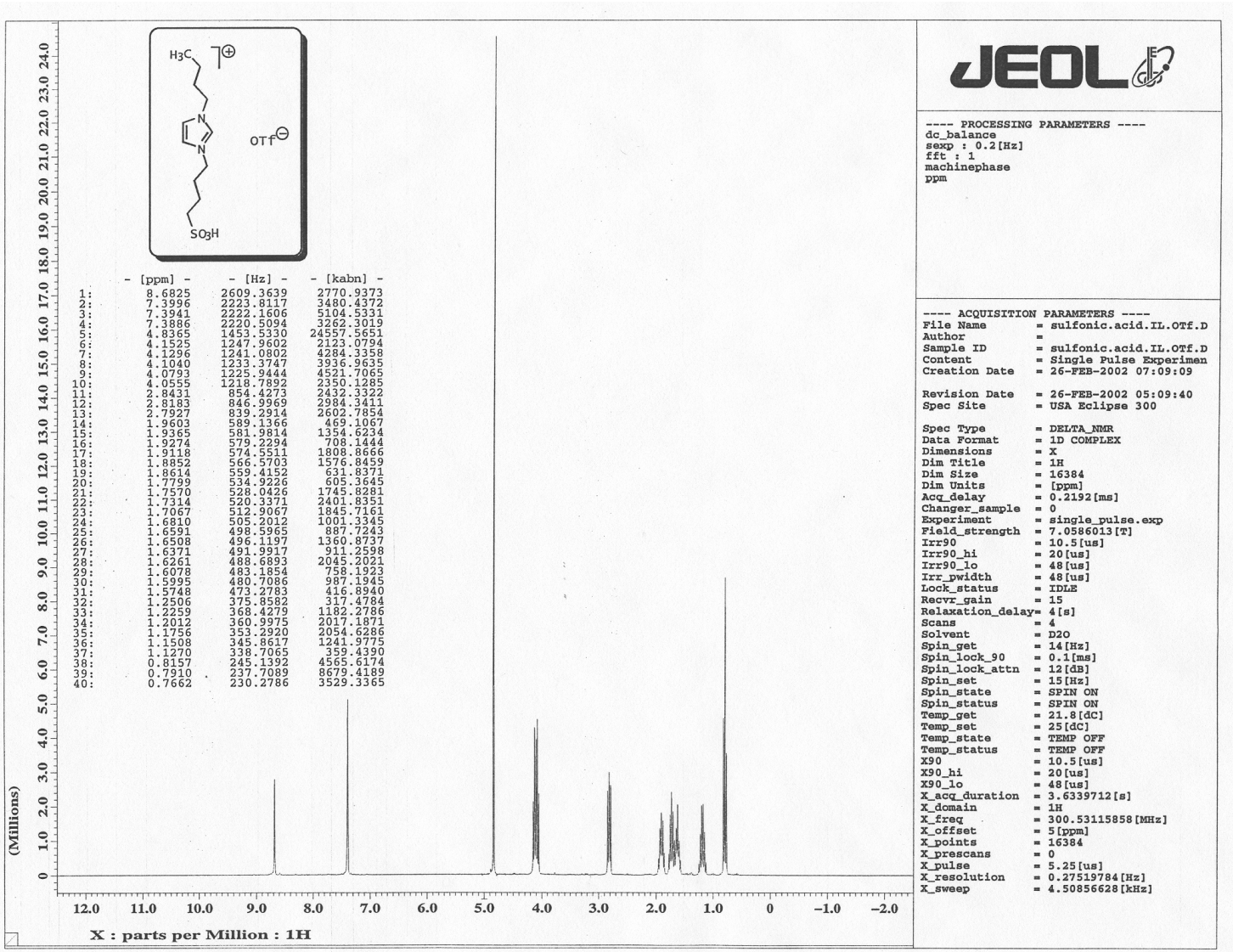

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21:	1.7570	528.0426	1745.8281
22:	1.7314	520.3371	2401.8351
23:	1.7067	512.9067	1845.7161
24:	1.6810	505.2012	1001.3345
25:	1.6591	498.5965	887.7243
26:	1.6508	496.1197	1360.8737
27:	1.6371	491.9917	911.2598
28:	1.6261	488.6893	2045.2021
29:	1.6078	483.1854	758.1923
30:	1.5995	480.7086	987.1945
31:	1.5748	473.2783	416.8940
32:	1.2506	375.8582	317.4784
33:	1.2259	368.4279	1182.2786
34:	1.2012	360.9975	2017.1871
35:	1.1756	353.2920	2054.6286
36:	1.1508	345.8617	1241.9775
37:	1.1270	338.7065	359.4390
38:	0.8157	245.1392	4565.6174
39:	0.7910	237.7089	8679.4189
40:	0.7662	230.2786	3529.3365

JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 0.2[Hz]
fft : 1
machinephase
ppm
```

```
---- ACQUISITION PARAMETERS ----
File Name          = sulfonic.acid.IL.OTf.D
Author             =
Sample ID          = sulfonic.acid.IL.OTf.D
Content            = Single Pulse Experimen
Creation Date      = 26-FEB-2002 07:09:09

Revision Date      = 26-FEB-2002 05:09:40
Spec Site          = USA Eclipse 300

Spec Type          = DELTA_NMR
Data Format        = 1D COMPLEX
Dimensions         = X
Dim Title          = 1H
Dim Size           = 16384
Dim Units          = [ppm]
Acq_delay          = 0.2192[ms]
Changer_sample     = 0
Experiment         = single_pulse.exp
Field_strength     = 7.0586013[T]
Irr90              = 10.5[us]
Irr90_hi           = 20[us]
Irr90_lo           = 48[us]
Irr_pwidth         = 48[us]
Lock_status        = IDLE
Recvr_gain         = 15
Relaxation_delay=  4[s]
Scans              = 4
Solvent            = D2O
Spin_get           = 14[Hz]
Spin_lock_90       = 0.1[ms]
Spin_lock_attn     = 12[dB]
Spin_set           = 15[Hz]
Spin_state         = SPIN ON
Spin_status        = SPIN ON
Temp_get           = 21.8[dC]
Temp_set           = 25[dC]
Temp_state         = TEMP OFF
Temp_status        = TEMP OFF
X90                = 10.5[us]
X90_hi             = 20[us]
X90_lo             = 48[us]
X_acq_duration     = 3.6339712[s]
X_domain           = 1H
X_freq             = 300.53115858[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 0
X_pulse            = 5.25[us]
X_resolution       = 0.27519784[Hz]
X_sweep            = 4.50856628[kHz]
```

JEOL
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2[Hz]
ffc : 1
machinephase
ppm
---- ACQUISITION PARAMETERS ----
File Name = triphenylphosphonium.I
Author =
Sample ID = triphenylphosphonium.I
Content = Single Pulse with Broa
Creation Date = 27-FEB-2002 00:29:46
Revision Date = 27-FEB-2002 06:02:40
Spec Site = USA Eclipse 300
Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 32768
Dim Units = [ppm]
Acq_delay = 51.5[us]
Changer_sample = 0
Experiment = single_pulse_dec
Field_strength = 7.0586013[T]
Irr90_hi = 10.5[us]
Irr90_lo = 20[us]
Irr_domain = 1H
Irr_pwidth = 48[us]
Lock_status = IDLE
Recvr_gain = 15
Relaxation_delay = 1[s]
Scans = 8192
Solvent = D2O
Spin_get = 17[Hz]
Spin_lock_90 = 0.1[ms]
Spin_lock_attn = 12[dB]
Spin_set = 15[Hz]
Spin_state = SPIN ON
Spin_status = SPIN ON
Temp_get = 21.4[dC]
Temp_set = 25[dC]
Temp_state = TEMP OFF
Temp_status = TEMP OFF
X90 = 8.5[us]
X90_hi = 8.5[us]
X90_lo = 36[us]
X_acq_duration = 1.73344272[s]
X_domain = 13C
X_freq = 75.57579184[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_pulse = 2.83333333[us]
X_resolution = 0.5769094[Hz]
X_sweep = 18.90359168[kHz]
- [ppm] - - [Hz] - - [kabn] -
1: 176.6367 13349.4615 1316.3410
2: 142.2096 10747.5999 22794.6905
3: 139.7516 10561.8351 13498.6817
4: 135.6142 10249.1502 1141.9978
5: 135.2860 10224.3431 44896.6010
6: 135.2478 10221.4585 45515.2753
7: 133.4692 10087.0386 92031.2013
8: 133.3394 10077.2312 91341.0655
9: 131.7974 9960.6955 1081.4701
10: 130.8203 9886.8511 1138.3581
11: 130.7058 9878.1974 1750.0998
12: 130.4005 9855.1210 84985.7725
13: 130.2326 9842.4290 85257.1455
14: 129.4234 9781.2766 82831.2493
15: 129.1257 9758.7772 1244.1827
16: 128.9654 9746.6621 966.7049
17: 127.6677 9648.5875 1541.2850
18: 125.7593 9504.3601 964.3046
19: 125.4387 9480.1299 73812.2473
20: 118.1258 8927.4507 27201.5205
21: 116.9808 8840.9142 25151.7937
22: 70.2178 5306.7670 907.6935
23: 68.8361 5202.3464 3742.9183
24: 60.2942 4556.7847 6041.8081
25: 50.5539 3820.6483 14940.2723
26: 50.3325 3803.9179 15120.5404
27: 48.0730 3633.1527 9893.7956
28: 47.1264 3561.6159 887.0365
29: 44.4928 3362.5822 924.7104
30: 27.0961 2047.8056 6300.7776
31: 24.3938 1843.5796 3297.8759
32: 24.1724 1826.8492 973.4828
33: 23.5770 1781.8503 918.0561
34: 20.7526 1568.3938 9719.0080
35: 20.5847 1555.7018 35324.9977
36: 20.0427 1514.7412 10188.3289
37: 17.9435 1356.0911 17647.8987
(Millions)
90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
176.6367
133.4692
133.3394
130.4005
130.2326
129.4234
125.4387
118.1258
116.9808
70.2178
68.8362
60.2942
50.5539
50.3325
48.0730
44.4928
27.0961
20.7526
20.5847
20.0427
17.9435
X : parts per Million : 13C

JEOL
---- PROCESSING PARAMETERS ----
1. DC_Balance
2. Hamming
3. FT 1
4. MachinePhase
5. PPM
6. Peak_Pick : 0[Hz] : 50[Hz] : Both
---- AQUISITION PARAMETERS ----
File Name = triphenylphosphonium.I
Author =
Sample_ID = triphenylphosphonium.I
Content = triphenylphosphonium.I
Creation_Date = 27-FEB-2002 00:35:36
Revision_Date = 26-FEB-2002 22:36:21
Spec_Site = USA Eclipse 300
Spec_Type = DELTA_NMR
Data_Format = 1D COMPLEX
Dimensions = X
Dim_Title = 1H
Dim_Size = 16384
Dim_Units = [ppm]
Acq_delay = 0.2192[ms]
Changer_sample = 0
Experiment = single_pulse.exp
Field_strength = 7.05860131[T]
Irr90 = 10.5[us]
Irr90_hi = 20[us]
Irr90_lo = 48[us]
Irr_pwidth = 48[us]
Lock_status = IDLE
Recvr_gain = 7
Relaxation_delay = 4[s]
Scans = 16
Solvent = D2O
Spin_get = 17[Hz]
Spin_lock_90 = 0.1[ms]
Spin_lock_attn = 12[dB]
Spin_set = 15[Hz]
Spin_state = SPIN ON
Spin_status = SPIN ON
Temp_get = 20.3[dC]
Temp_set = 25[dC]
Temp_state = TEMP OFF
Temp_status = TEMP OFF
X90 = 10.5[us]
X90_hi = 20[us]
X90_lo = 48[us]
X_acq_duration = 3.63397121[s]
X_domain = 1H
X_freq = 300.53115858[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_pulse = 5.25[us]
X_resolution = 0.27519784[Hz]
X_sweep = 4.50856628[kHz]
(Millions)
X : parts per Million : 1H