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A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

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In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Preparation of 3-Oxocyclohex-1-ene-1-carbonitrile

A.

B.
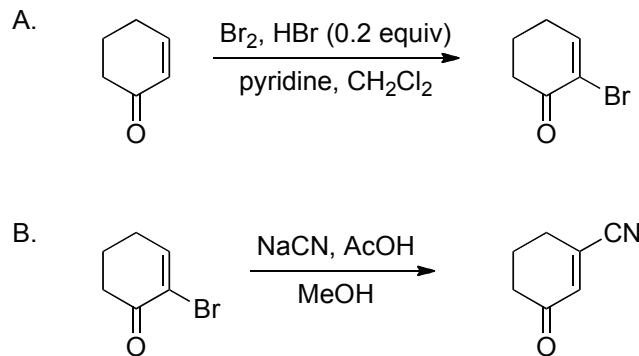
Submitted by Jesus Armando Lujan-Montelongo and Fraser F. Fleming.[1]
Checked by David Hughes.

1. Procedure

A.   2-Bromo-2-cyclohexen-1-one. A 3-necked, 500-mL, round-bottomed flask, equipped with a magnetic stir bar (PTFE-coated, oval, 4 cm), is charged with cyclohex-2-enone (15.0 g, 156 mmol, 1.0 equiv) and CH_2Cl_2 (150 mL) (Note 1). The center neck is fitted with a 25-mL addition funnel equipped with a gas inlet adapter connected to a nitrogen line and gas bubbler. One outer neck is sealed with a glass stopper; the other neck is capped with a rubber septum through which a thermocouple probe is inserted (Note 2). The flask is immersed in a dry ice/acetonitrile bath and stirring is begun. After the internal temperature reaches -45 °C, HBr (48% aq. solution, 3.6 mL, 32 mmol, 0.2 equiv) is added dropwise via syringe through the septum in 1 min. The addition funnel is charged with neat Br_2 (9.0 mL, 175 mmol, 1.1 equiv), which is then added dropwise until the orange-red color indicative of excess bromine persists (Note 3). After 10 min, the addition funnel is replaced by a clean 125-mL addition funnel charged with pyridine (25.5 mL, 316 mmol, 2.0 equiv), which is then added dropwise over 15 min (Note 4). After the addition, the cold bath is removed and the dropping funnel is replaced by a reflux condenser equipped with a gas inlet adapter, which is connected to a nitrogen line and gas bubbler. The mixture is allowed to warm to room temperature over 30 min, then heated to reflux (43 °C) for 1 h using a heating mantle. After cooling to room temperature the flask contents are transferred to a 1-L separatory funnel, the

reaction flask is washed with CH_2Cl_2 (50 mL), and the additional solution is added to the separatory funnel. The mixture is washed with an aqueous solution of sodium thiosulfate (0.7 M, 150 mL). The organic phase is separated and the aqueous phase extracted with CH_2Cl_2 (50 mL). The combined organic layers are washed with 1M HCl (100 mL). The organic layer is removed and the acidic, aqueous phase is extracted with CH_2Cl_2 (50 mL). The combined organic fraction is washed with water (50 mL), brine (50 mL), and then filtered through a bed of Na_2SO_4 (50 g) into a tared 1-L round-bottomed flask. The solution is concentrated by rotary evaporation (40 °C, 200 mmHg) to about 60 mL (84 g), then hexanes (100 mL) are added and the concentration continued to dryness (Note 5). Vacuum drying (10 mmHg) to constant weight (1.5 h) affords crude 2-bromo-2-cyclohexenen-1-one (26.7 g, 98% yield) as a slightly yellow powder. The crude 2-bromo-2-cyclohexen-1-one is suitable for the cyanation-elimination reaction without purification (Notes 6 - 8).

> *Caution! Sodium cyanide and HCN are extremely toxic. The experimentalist should use sufficient personal protection and only handle sodium cyanide solid and solutions in a well-ventilated fume hood. The aqueous waste solutions containing cyanide should be treated with excess bleach before disposal.[2] Good housekeeping is essential - all spills around balances or work areas should be immediately cleaned up and the area washed down with bleach.*

B. *3-Oxocyclohex-1-enecarbonitrile.* A 500-mL round-bottomed flask is charged with MeOH (120 mL) (Note 9), crude 2-bromo-2-cyclohexen-1-one (18.6 g, 106 mmol, 1.0 equiv), and a magnetic stir bar (PTFE-coated, oval, 3 cm). The flask is capped with a rubber septum and inserting an 18-gauge needle through the septum provides a nitrogen inlet. A thermocouple probe is also inserted through the septum (Note 2). The reaction flask is immersed in a tap-water bath and then neat AcOH (6.5 mL, 114 mmol, 1.1 equiv) is added dropwise to the stirred solution via syringe over 1 min. After 5 min, the septum is briefly removed, solid NaCN (5.68 g, 116 mmol, 1.1 equiv) is added in one portion, and the septum is replaced (Note 10). After 10 min a second portion of NaCN (2.73 g, 56 mol, 0.5 equiv) is added, followed, after another 10 min, by a third portion of NaCN (1.25 g, 26 mmol, 0.25 equiv), replacing the septum after each addition. After 20 min (Note 11), the water

bath is replaced with an ice-water bath. Once the flask contents equilibrate to 5 °C, the septum is removed, and an aqueous solution of Na_2CO_3 (0.5 M, 100 mL) is added. After 1 min, solid NaCl (10 g) is added and the mixture is stirred for 5 min. The flask contents are transferred to a 1-L separatory funnel, the reaction flask is washed with EtOAc (100 mL), and the additional solution is added to the separatory funnel. The organic phase is separated and the aqueous phase is extracted with EtOAc (3 x 100 mL). The combined organic phase is washed with 5 °C aqueous NaOH (1M, 100 mL) (Note 12) and then washed with brine (2 x 50 mL). The organic phase is dried by filtering through a bed of Na_2SO_4 (50 g) into a tared 1-L round-bottomed flask. The solvent is removed by rotary evaporation (40 °C, 10 mmHg) to afford an orange oil (12.6 g), which is purified by column chromatography on Florisil (Notes 13 and 14) to afford 3-oxocyclohex-1-enecarbonitrile (8.07 g, 63% yield) as a pale yellow oil spectrally identical to material previously reported (Note 15).[3]

2. Notes

1. The following reagents and solvents were used as received for Step A: cyclohex-2-enone (Sigma-Aldrich, 95+%), HBr (48% aq., Sigma-Aldrich), CH_2Cl_2 (Fischer Certified ACS, stabilized), Br_2 (Sigma-Aldrich, reagent grade), and pyridine (Sigma-Aldrich, ACS reagent, >99.0%).

2. The internal temperature was monitored using a J-Kem Gemini digital thermometer with a Teflon-coated T-Type thermocouple probe (12-inch length, 1/8 inch outer diameter, temp range –200 to +250 °C).

3. Bromine is extremely corrosive and an irritant and must be transferred within a well-ventilated fume hood. Dropwise addition of bromine at intervals of one drop every 2-3 sec, slowing to one drop every 5 sec near the end point, gave the best results. The entire addition time was 40 min. The internal temperature was maintained at –45 to –48 °C. An excess of three drops of bromine is typically added after the end point to ensure the orange-red color indicative of excess bromine persists. Unreacted starting material carries through to the next step and cannot be removed in the chromatographic purification of the nitrile product of step B.

4. Pyridine was added at a rate of about 1 drop/sec with the temperature rising to –39 °C. The reaction was monitored by ^{1}H NMR by removing a 0.1 mL reaction aliquot and quenching into 0.5 mL EtOAc/0.5 mL 0.7M sodium thiosulfate solution, separating the

organic layer, filtering through a cotton plug, and concentrating to dryness. Diagnostic ^{1}H NMR resonances: starting material, δ: 6.01 (dt, J = 2.0, 10.3 Hz, 1 H) and 7.03 (dt, J = 4.3, 10.3, 1 H); product, δ: 7.42 (t, J = 4.5 Hz, 1 H).

5. The flush with hexanes results in crystallization during the concentration, which provides a product with greater stability. The solid obtained by concentration of the dichloromethane solution was found to be less stable.

6. The crude material was 95% pure based on GC analysis (t_R = 9.7 min; conditions: Agilent DB35MS column; 30 m x 0.25 mm; initial temp 60 °C, ramp at 20 °C/min to 280 °C, hold 15 min). The submitters carried out a recrystallization of 18.0 g of 2-bromo-2-cyclohexen-1-one by dissolution in 25 mL of boiling EtOAc, filtration through a pre-wetted funnel with filter paper, followed by the addition of hot hexane (~4 mL) to induce turbidity, cooling, filtration on a Büchner funnel, and drying for 20 min at 0.01 mmHg, providing 13.3 g of white crystals (73% yield, mp 74–75 °C).[4] The checker purified the product as follows. To a 100-mL round-bottomed flask equipped with a 2-cm oval PTFE-coated magnetic stir bar was added crude 2-bromo-2-cyclohexen-1-one (7.80 g) and *t*-BuOMe (10 mL). The slurry was stirred for 5 min. The flask was fitted with a 25-mL addition funnel through which was added *n*-heptane (10 mL) over a 10 min period. The slurry was stirred for 20 min at room temperature, then filtered and washed with *n*-heptane (10 mL) to afford 2-bromo-2-cyclohexen-1-one as an off-white solid (5.45 g, 70% recovery, mp 72–74 °C, >99% pure by GC).

7. 2-Bromo-2-cyclohexen-1-one has the following spectroscopic properties: ^{1}H NMR (400 MHz, CDCl$_3$) δ: 2.04–2.10 (m, 2 H), 2.43–2.47 (m, 2 H), 2.60–2.64 (m, 2 H), 7.42 (t, J = 4.5 Hz, 1 H); ^{13}C NMR (100 MHz, CDCl$_3$) δ: 22.8, 28.5, 38.5, 124.0, 151.3, 191.4; IR (neat): 3043, 1680, 1598 cm^{-1}; HRMS (ESI) calculated for C$_6$H$_7$BrO 196.9572; found 196.9570 (M+Na)$^+$; GC-MS *m/z* (rel intensity): 176 (M$^+$, 78), 174 (M$^+$, 77), 148 (87), 146 (88), 135 (24), 133 (24), 120 (38), 118 (38), 67 (100), 55 (42).

8. The checker found both crude and recrystallized 2-bromo-2-cyclohexen-1-one to be unstable at room temperature, decomposing within 3 days to a purple gum with release of gas. Material stored in the freezer darkened but remained unchanged by NMR for a period of 2 weeks.

9. The following reagents and solvents were used as received for Step B: methanol (Fisher Optima, 99.9%), HOAc (Fisher Certified ACS,

100.0%), NaCN (Sigma-Aldrich, ACS certified, 95+%), EtOAc (Fisher, ACS reagent), Florisil (Sigma-Aldrich, 100-200 mesh), hexanes (Fisher, ACS reagent, >98.5%), methyl t-butyl ether (Sigma-Aldrich, >98.5%).

10. The temperature rose as follows after the NaCN additions: #1, 18 to 28 °C; #2, 25 to 28 °C; #3, 24 to 25 °C.

11. Progress of the reaction was monitored by TLC (1:1 MTBE/hexanes) and visualized with UV light; R_f = 0.4 for product and 0.5 for starting material. The checker found the reaction achieved >95% completion by the current protocol. The submitters suggest that if complete conversion is not achieved, an additional portion of NaCN (0.25 equiv) and AcOH (3.2 mL) can be added and the reaction checked again after 20 min. If necessary, the water bath temperature can be raised to 50–55 °C in order to facilitate complete conversion.

12. Washing the organic phase with NaOH is designed to purge the solution of residual HCN. These precautions minimize the potential for contact with HCN during removal of solvent on a rotary evaporator that must be vented to, or located in, a fume hood. Residual NaCN is removed in the aq. washes, which should be treated with bleach prior to disposal.

13. Column chromatography on silica gel causes significant adsorption of the oxonitrile resulting in a low yield. However, the submitters found that 2 g of cyclohex-2-enone provided crude keto nitrile that was readily purified by radial chromatography (4 mm SiO$_2$ plate, EtOAc/hexanes as eluent, 1:19) without significant adsorption. Concentration on a rotary evaporator provided pure 3-oxocyclohex-1-ene-carbonitrile (1.77 g, 70% yield over two steps).

14. Florisil (325 g) was wet-packed in a 7-cm diameter column using MTBE:hexanes (1:3) with a 0.5 cm bed of sand topping the column. The crude oil was loaded neat and rinsed onto the column with 2 x 5 mL MTBE. Elution was carried out with 1 L MTBE:hexanes (1:3), 1 L MTBE:hexanes (1:2), and 1 L of MTBE:hexanes (2:3). Column flow was 40 mL/min and 50 mL fractions were collected. Product elution was followed by TLC as indicated in Note 11. Fractions 33-50 were combined and concentrated by rotary evaporation (40 °C bath, 100 mmHg to 10 mmHg), then dried under vacuum (10 mmHg for 2 h) to constant weight to afford 7.20 g of a pale yellow oil. Purity by GC was 95 % (same conditions as Note 6, t_R = 8.9 min). Fractions 30-32 and 51-60 were combined and likewise concentrated to 1.50 g. Purity by GC was 78%. These fractions were re-chromatographed using 100 g of Florisil, eluting with 300 mL

MTBE:hexanes (1:3) and 600 mL MTBE:hexanes (1:2), collecting 25 mL fractions. Fractions 22-33 were combined and concentrated by rotary evaporation (40 °C bath, 100 mmHg to 10 mmHg), then vacuum dried to constant weight to afford 0.87 g of product. Purity by GC was 92%. The rich cuts from the two chromatographies were combined to afford product (8.07 g, 63% yield, GC purity 95%) as a pale yellow oil.

15. 3-Oxocyclohex-1-enecarbonitrile has the following spectroscopic properties: ^{1}H NMR (400 MHz, CDCl$_3$) δ: 2.10–2.17 (m, 2 H), 2.49–2.53 (m, 2 H), 2.57 (td, J = 6.0, 2.0 Hz, 2 H), 6.52 (t, J = 2.0 Hz, 1 H); ^{13}C NMR (100 MHz, CDCl$_3$) δ: 22.3, 27.8, 37.4, 117.2, 131.2, 138.9, 196.5. IR (neat) 3059, 2232, 1682, 1606 cm^{-1}; HRMS calculated for C$_7$H$_7$NO 144.0420; found 144.0414 (M+Na)$^+$. GC-MS m/z (rel intensity): 121 (M$^+$, 68), 93 (100), 66 (27), 65 (38), 64 (30). After 4 months of storage at 4 °C the submitters found the sample underwent modest discoloration from light yellow to orange, although no loss of sample integrity was observed by ^{1}H NMR and there was no change in reactivity.

Safety and Waste Disposal Information

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3. Discussion

Cyclic oxoalkenenitriles juxtapose three orthogonal functionalities capable of selective functionalization; an olefin, a ketone, and a nitrile functionality.[5] 3-Oxocycloalkenecarbonitriles, in particular, are excellent

scaffolds for diversity oriented synthesis,[6] feature strategically in syntheses,[7] and as precursors for mechanistic studies.[8] The electron deficient olefin in 3-oxocyclohex-1-ene-1-carbonitrile (Scheme 1, **1**) is an excellent participant in [2+2][9] (**1** → **2** and **1** → **3**) and [4+2][10] cycloadditions (**1** → **4**), and in conjugate additions (**1** → **5**).[11] Sequential addition of two Grignard reagents to 3-oxocyclohex-1-ene-1-carbonitrile (**1**) allows stepwise 1,2-1,4-additions to afford the extremely versatile *C*-magnesiated nitrile **5.**[11] Stereoselective annulations[12] provide access to bicyclic and tricyclic nitriles **6**[11] and **7**,[6] whereas complementary alkylation strategies access nitriles **8** and **9** bearing diastereomeric, quaternary centers,[13] and allow *N*-alkylation to enamides **10**.[14]

Scheme 1. Synthetic Applications of 3-Oxocyclohex-1-enecarbonitrile (**1**)
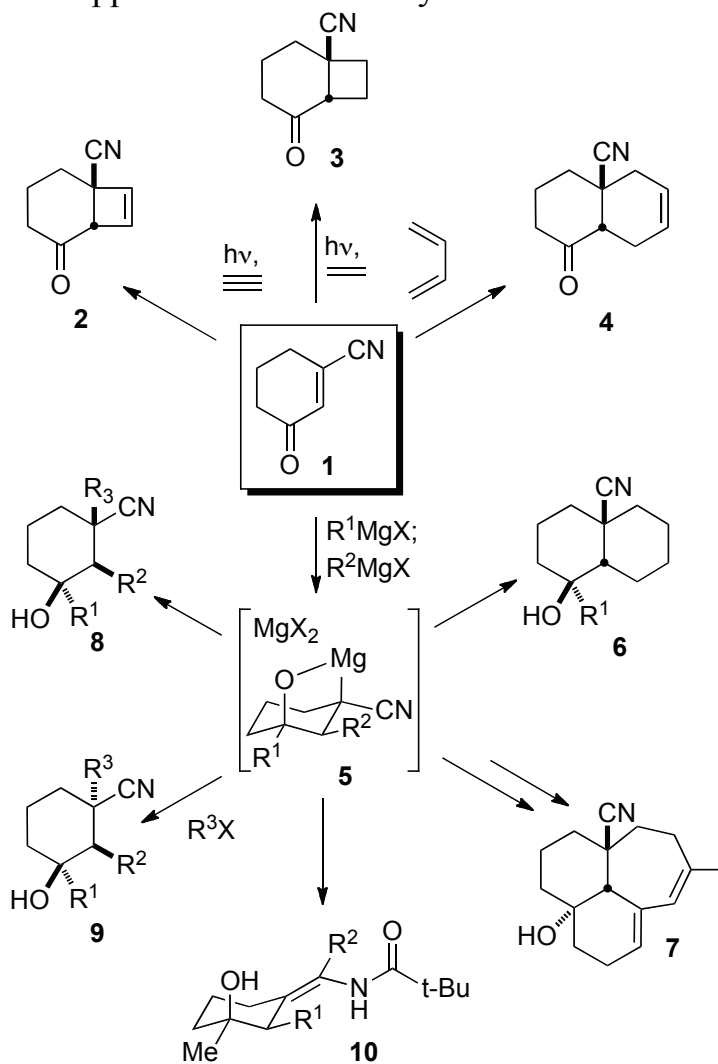
The value of 3-oxocyclohex-1-ene-1-carbonitrile (**1**) has stimulated several syntheses: cyanide addition to 1,3-cyclohexandione monoethylene

ketal,[15] Et$_2$AlCN addition to 3-methoxycyclohexenone,[16] or to cyclohexenone followed by periodinane oxidation,[17] oxidative transposition of an allylic cyanohydrin,[18] and allylic oxidation of cyclohexenecarbonitrile with PhI(OAc)$_2$ and t-BuOOH,[19] or with CrO$_3$ and 3,5-dimethylpyrazole.[3] Among strategies for synthesizing **1**,[3,14-19] and related sequences employed in total synthesis campaigns,[7] the bromination-cyanation of cyclic enones[20] is conspicuous for efficiency, cost, and operational simplicity (Scheme 2). Formation of 2-bromocyclohex-2-enone (**12**) from cyclohexenone (**11**) is fast and virtually quantitative. The subsequent conjugate addition of cyanide provides an intermediate ketone **13** from which dehydrohalogenation is readily achieved simply through modest heating. The synthesis of 3-oxocyclohex-1-ene-1-carbonitrile (**1**) is very expedient and inexpensive. Operationally, the reaction uses standard glassware and affords gram quantities of 3-oxocyclohex-1-ene-1-carbonitrile (**1**) from cyclohexenone in 2-steps that can be completed in one day.

Scheme 2. Synthesis of 3-Oxocyclohex-1-enecarbonitrile (**1**)
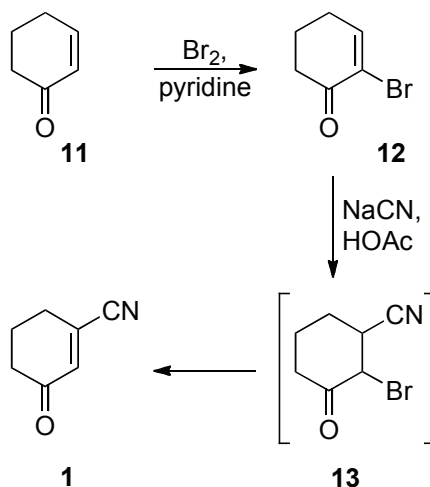
Employing the bromination-cyanation sequence with the homolog cycloheptenone (**14**) affords the seven-membered 3-oxocyclohept-1-ene-1-carbonitrile (Scheme 3, **16**).[8b,18,21] The bromination of cycloheptenone (**14**) parallels the bromination of cyclohexenone (**11**), although the elimination of the intermediate dibromide requires heating the reaction mixture for 30 min at 70 °C to form 2-bromocyclohept-2-enone (**15**). Control over the temperature required for the elimination of HBr during the formation of 2-bromocyclohept-2-enone (**15**) is critical. The temperature was conveniently controlled using microwave irradiation,[22] which reproducibly affords very

pure 2-bromocyclohept-2-enone. Subsequent cyanation-elimination affords 3-oxocyclohept-1-enecarbonitrile (**16**) in 71% yield over two steps. The resulting 3-oxocyclohept-1-enecarbonitrile functionality has been strategically employed in total synthesis[20] and is a valuable partner for sequential 1,2-1,4-addition-alkylations.[21]

Scheme 3. Synthesis of 3-Oxocyclohept-1-ene-1-carbonitrile (**16**)
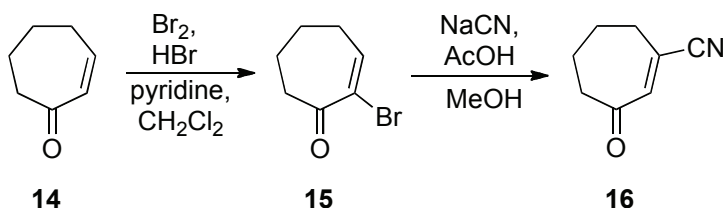
Collectively, the bromination-cyanation of cyclic enones provides efficient, cost-effective syntheses of 3-oxocycloalkene-1-carbonitriles. The syntheses are rapid and provide access to functionalized building blocks ideally suited for synthetic applications.

1. Department of Chemistry, Duquesne University, Pittsburgh, PA 15282-1530, email: flemingf@duq.edu. This work was supported by the National Science Foundation (CHE 1111406) and in part by CONACYT (J. A. L.-M.). Drew Davic is thanked for assistance in performing GCMS.
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22. A Biotage® microwave reactor (Model: Initiator) and a 20 mL reaction tube was used in a reaction employing 1.8 g of cyclohept-2-enone. The radiation absorption parameter was set to NORMAL, with the apparatus initially recording a slight increase of pressure (2 - 3 Psi).

Appendix
Chemical Abstracts Nomenclature; (Registry Number)

2-Bromo-2-cyclohexen-1-one; (50870-61-6)
Cyclohex-2-enone; (930-68-7)
3-Oxocyclohex-1-enecarbonitrile; (CAS# 25017-78-1)

Fraser Fleming earned his B. Sc. (Hons.) at Massey University, New Zealand, in 1986 and a Ph. D. under the direction of Edward Piers at the University of British Columbia, Canada, in 1990. After postdoctoral research with James D. White at Oregon State University he joined the faculty at Duquesne University, Pittsburgh, in 1992. His research interests lie in stereochemistry and organometallics, particularly as applied to alkenenitrile conjugate additions and metalated nitrile alkylations.

Jesus Armando Lujan-Montelongo, a native of Queretaro, Mexico, completed his BS at the National University Autonomous of Mexico (UNAM) in 2003. He continued his studies at UNAM, earning his Ph. D. in Organic Chemistry in 2005 under the direction of Jose G. Avila-Zarraga. After postdoctoral studies with Luis Miranda at the Institute of Chemistry (UNAM) developing free-radical based synthetic methods, he joined Fraser Fleming at Duquesne University in Pittsburgh, where he is working on nitrile-based methodology. His research interests lie in the total synthesis of natural products and in organometallic chemistry.
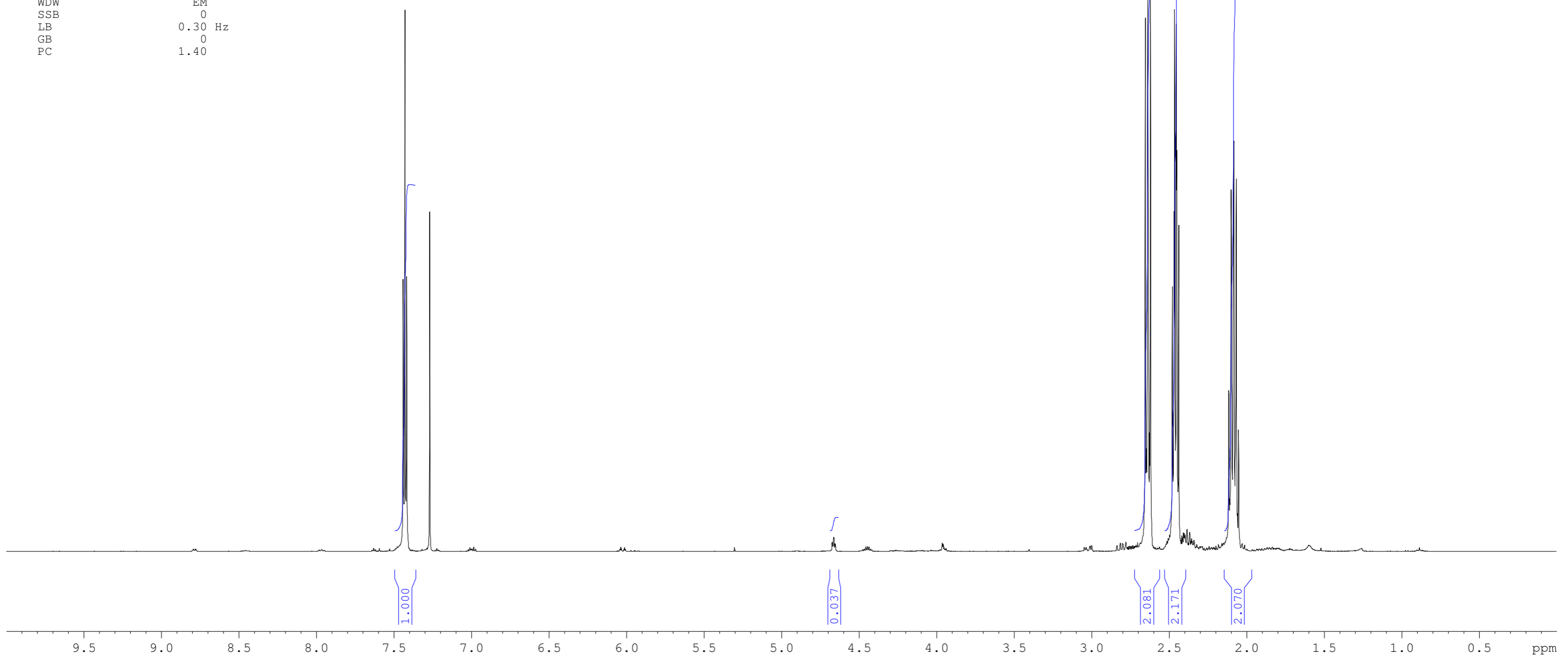
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32077-225
crude
nmr400b h-1

CRUDE

32077-221/2 hughesda
32077-221
recrystallized
nmr400b h-1
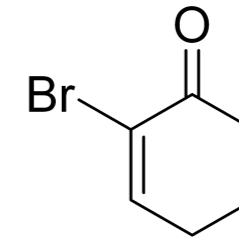
Recrystallized
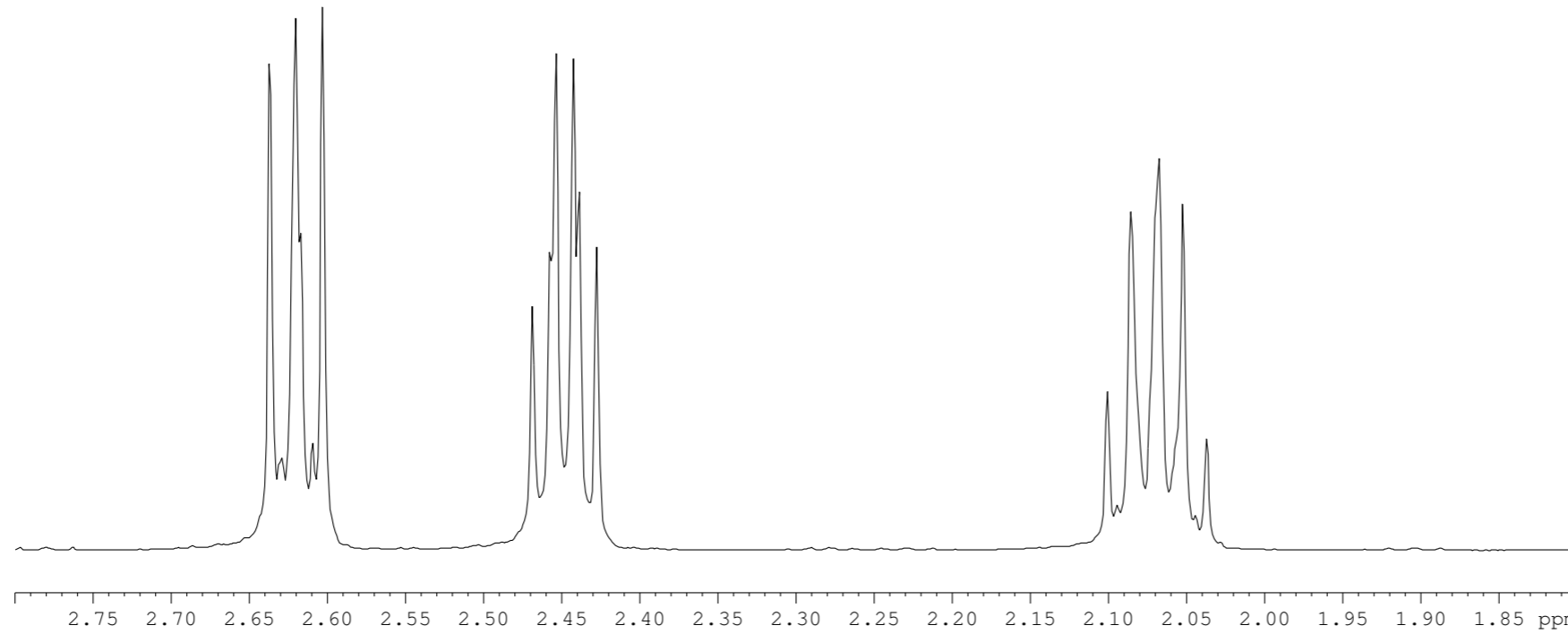
Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]	Annotation
1	7.4286	2973.8915	10785416.81	
2	7.4175	2969.4478	27688728.56	
3	7.4063	2964.9641	13831562.56	
4	7.2699	2910.3591	6304228.38	
5	2.6366	1055.5101	27032485.94	
6	2.6292	1052.5476	4909354.12	
7	2.6200	1048.8646	25808904.94	
8	2.6173	1047.7837	17152668.81	
9	2.6091	1044.5010	5377852.00	
10	2.6029	1042.0190	30177629.00	
11	2.4685	988.2146	13544156.19	
12	2.4570	983.6108	16566205.19	
13	2.4536	982.2497	23869519.94	
14	2.4423	977.7260	27322772.00	
15	2.4387	976.2848	18447514.81	
16	2.4274	971.7611	12395801.06	
17	2.1005	840.8932	7182883.44	
18	2.0942	838.3711	2510820.69	
19	2.0852	834.7681	18794757.00	
20	2.0675	827.6823	20460049.06	
21	2.0521	821.5172	19243814.31	
22	2.0442	818.3546	1963757.62	
23	2.0370	815.4722	6199018.81	

1.000

2.189
2.219

2.229

NAME   32077-221
EXPNO   3
PROCNO   1
Date_   20130203
Time   13.13
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zgdc
TD   65536
SOLVENT   CDCl3
NS   1365
DS   4
SWH   26315.789 Hz
FIDRES   0.401547 Hz
AQ   1.2452340 sec
RG   8192
DW   19.000 usec
DE   6.50 usec
TE   300.0 K
D1   0.10000000 sec
D11   0.03000000 sec
TD0   40

======== CHANNEL f1 ========
NUC1   13C
P1   3.50 usec
PL1   0.00 dB
PL1W   31.90095711 W
SFO1   100.6741319 MHz

======== CHANNEL f2 ========
CPDPRG2   waltz16
NUC2   1H
PCPD2   80.00 usec
PL2   120.00 dB
PL12   17.00 dB
PL2W   0.00000000 W
PL12W   0.16438942 W
SFO2   400.3320017 MHz
SI   32768
SF   100.6630422 MHz
WDW   EM
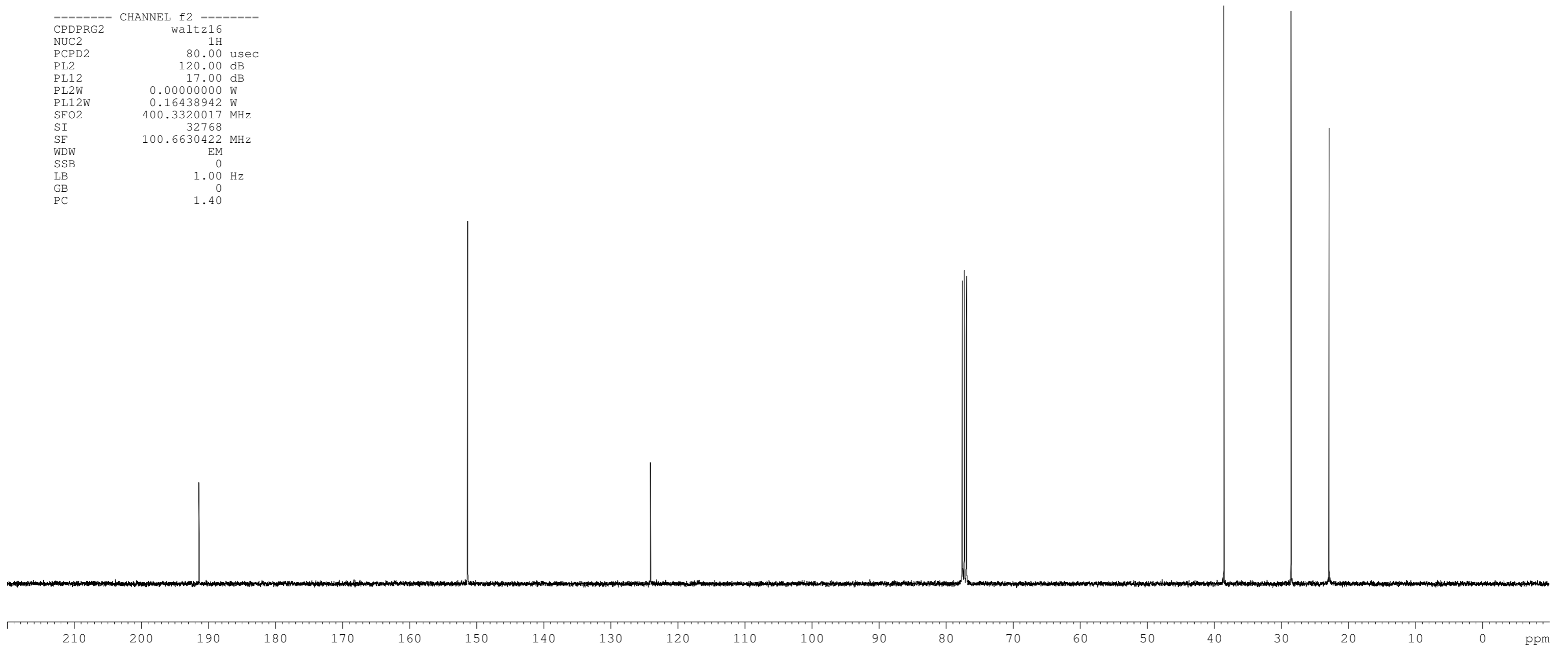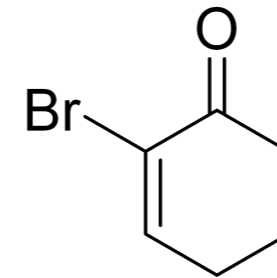SSB   0
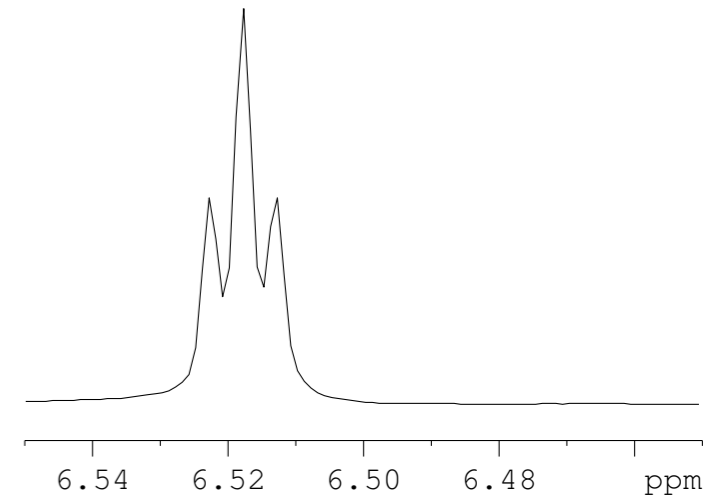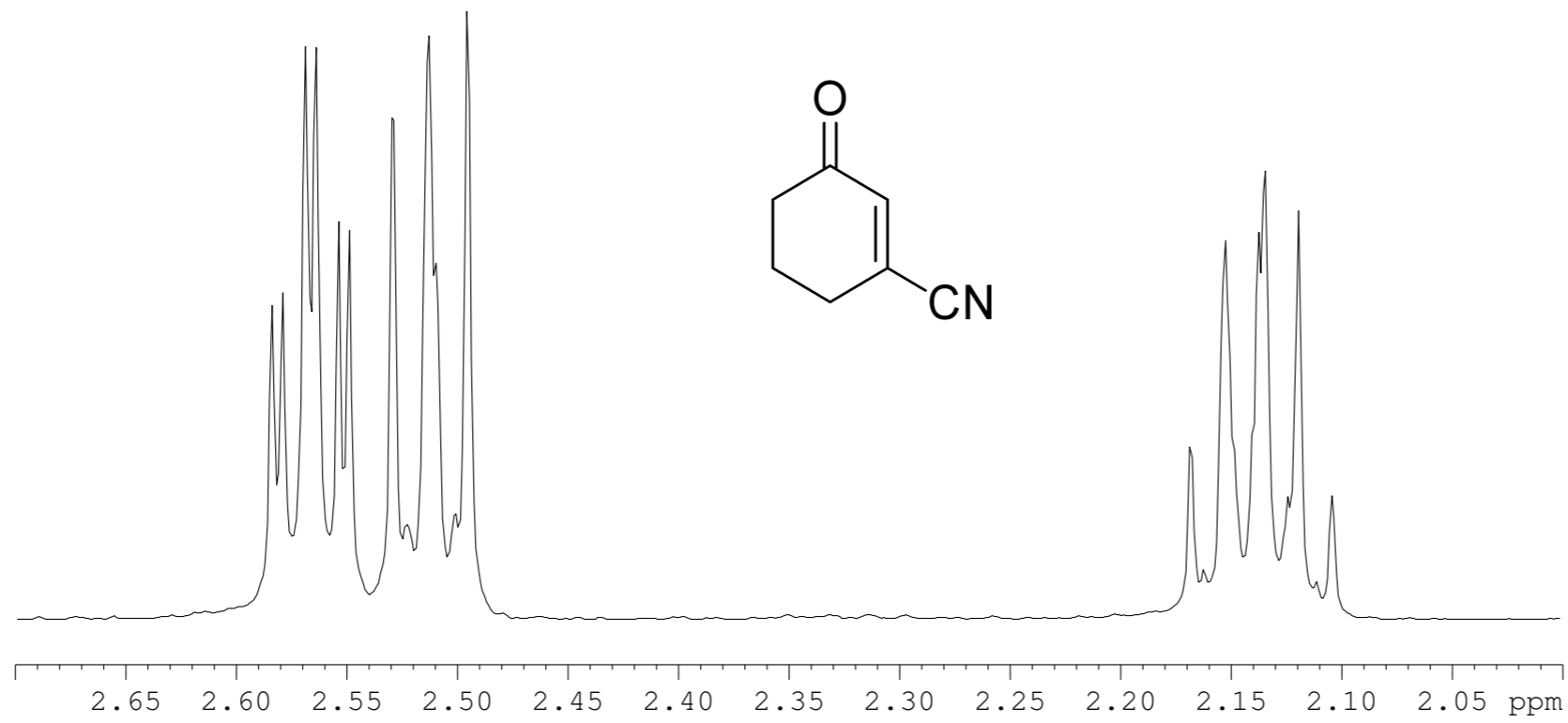LB   1.00 Hz
GB   0
PC   1.40

Peaks: 151.33, 124.04, 77.55, 77.23, 76.91, 38.51, 28.52, 22.84

32077-226/2 hughesda
32077-226
fr 38
nmr400b h-1

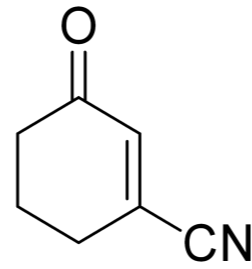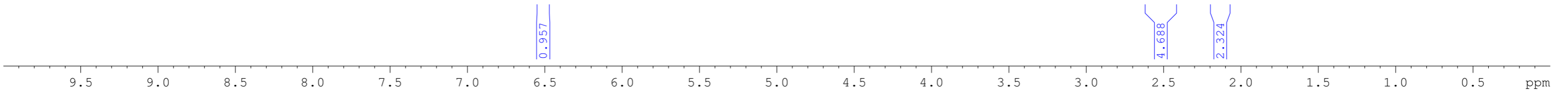| Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]	Annotation
1	7.2699	2910.3591	13756674.06	
2	6.5226	2611.1925	10605472.94	
3	6.5177	2609.2309	20305939.44	
4	6.5129	2607.3093	9112987.75	
5	2.5838	1034.3727	11331206.44	
6	2.5789	1032.4110	9452202.19	
7	2.5687	1028.3277	20653384.31	
8	2.5638	1026.3661	17295328.44	
9	2.5537	1022.3227	14368453.19	
10	2.5488	1020.3611	10010729.44	
11	2.5291	1012.4746	18114545.19	
12	2.5226	1009.8725	3462617.81	
13	2.5129	1005.9893	20097770.88	
14	2.5098	1004.7482	12418825.56	
15	2.5009	1001.1853	3776546.31	
16	2.4953	998.9435	21950509.62	
17	2.1681	867.9555	6246009.06	
18	2.1624	865.6736	1833303.06	
19	2.1525	861.7103	13670781.00	
20	2.1373	855.6253	13989336.88	
21	2.1348	854.6245	15417191.81	
22	2.1241	850.3410	4448535.81	
23	2.1194	848.4594	14760912.75	
24	2.1113	845.2167	1405541.75	
25	2.1042	842.3744	4480568.81	

NAME   32077-226
EXPNO   3
PROCNO   1
Date_   20130309
Time   11.52
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zgdc
TD   65536
SOLVENT   CDCl3
NS   2427
DS   4
SWH   26315.789 Hz
FIDRES   0.401547 Hz
AQ   1.2452340 sec
RG   8192
DW   19.000 usec
DE   6.50 usec
TE   300.0 K
D1   0.10000000 sec
D11   0.03000000 sec
TD0   40

======== CHANNEL f1 ========
NUC1   13C
P1   3.50 usec
PL1   0.00 dB
PL1W   31.90095711 W
SFO1   100.6741319 MHz

======== CHANNEL f2 ========
CPDPRG2   waltz16
NUC2   1H
PCPD2   80.00 usec
PL2   120.00 dB
PL12   17.00 dB
PL2W   0.00000000 W
PL12W   0.16438942 W
SFO2   400.3320017 MHz
SI   32768
SF   100.6630380 MHz
WDW   EM
SSB   0
LB   1.00 Hz
GB   0
PC   1.40

196.51

138.87
131.17

117.16

77.55
77.43
77.23
76.91

37.44

27.83
22.27