Example Experimental Sections: (Explanatory notes are indicated by bold letters; **[a]**, **[b]**, **[c]**, **[d]**, etc.)

Example 1: General Section

General. **[a]** Unless otherwise noted, reagent solutions were added with a syringe. Chromatography was carried out with Merck 60 230-400 mesh silica gel according to the procedure described by Still[1] unless otherwise noted. Reactions and chromatography fractions were analyzed with Analtech 250 micron silica gel GF plates. When an R_f is reported for a compound, the solvent that was used was the chromatography solvent unless otherwise indicated. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Ether and tetrahydrofuran (THF) were distilled under N_2 from Na/benzophenone immediately prior to use. **[b]** Toluene, xylenes, triethylamine (Et_3N) and diisopropylamine (i-Pr_2NH) were distilled from CaH_2 and stored over 3 or 4 Å molecular sieves. Methylene chloride (CH_2Cl_2) was distilled from P_2O_5 immediately prior to use. Dimethyl sulfoxide (DMSO) **[c]** was distilled from BaO and stored over 3 Å molecular sieves. The concentration of commercially available solutions of *n*-butyllithium in hexanes was periodically checked by titration with diphenylacetic acid.[2] Unless otherwise specified, extracts were dried over $MgSO_4$ and solvents were removed with a rotary evaporator at aspirator pressure. **[d]** Unless otherwise indicated, IR spectra were of thin films on NaCl plates and NMR were measured in $CDCl_3$. **[e]** **[f]** In some cases Distortionless Enhancement by Polarization Transfer (DEPT)[3] was used to assign the ^{13}C NMR resonances as CH_3, CH_2, CH, or C. **[g]**

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[a] Details that are common to all (or a large number) of procedures should be reported in a "General" section, placed at the beginning of the experimental section of the report or paper. The title should be in bold face print and indented (use the "tab" key with a word processor). If a word processor is not used, use "wiggly underlining" to indicate bold face type.

[b] Describe how important solvents were purified and stored, if pertinent.

[c] Use abbreviations and acronyms that are approved by the journal for which you are writing. The *Journal of Organic Chemistry* publishes a list of approved abbreviations and acronyms annually with its "Guidelines for Authors."

[d] The General Section should contain descriptions of common procedures, like how you dried and concentrated ether or CH_2Cl_2 extracts. This can save an enormous number of lines of print in a thesis or journal.

[e] Specify the solvent normally used for IR and NMR spectra in the General section. In addition, if you always used the same NMR spectrometer, you should give the field strength here. Remember that it is important to give the field strength somewhere for every ^1H NMR spectrum you report. This is because complex multiplets like dd, dt, ddd, etc. may actually have different appearances at different field strengths.

[f] It used to be common procedure to give the brand names of instruments used. However, to save space, journals now discourage this practice unless the instrument is a highly specialized one.

[g] Give literature references for any unusual analytical procedures that were used.

When $^{13}C\{H\}$ NMR spectra [h] [i] were obtained with mixtures of stereoisomers, some of the resonances overlapped. [j] Therefore, the correct number of resonances may not be listed. Also, when carbons are equivalent (eg. ortho and meta protons of a benzyl group) no special notation is used. Gas chromatography was performed with a 4% Carbowax 20M on 60/80 mesh Chromosorb G column.

Example 2: Preparation of a Previously Unknown Compound

1-[1-Oxo-5-(phenylmethoxy)pentyl]pyrrolidine (9). [k] [l] To 19.8 g (95.0 mmol) of 5-(phenylmethoxy)pentanoic acid[4] in 200 mL [m] of dry CH_2Cl_2 was added 17.0 g (105 mol) [n] of 1,1'-carbonyldiimidazole in portions over 10 min (vigorous evolution of CO_2!). [o] The flask was immersed in a cool water bath after the

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[h] Note that "spectrum" is singular and "spectra" is plural. Similarly, "datum" is singular and "data" is plural. Thus, you should not say that "the ^1H NMR spectra was measured. . ." or that "the data was consistent with. . ."

[i] The designation {H} following C means that spectra were determined while simultaneously decoupling at the proton resonance frequency.

[j] Note the form for describing the NMR nucleus: ^1H NMR, ^{13}C NMR, ^{19}F NMR, etc. Remember that you do not "run an NMR", but rather a "NMR spectrum." Also, "Bruker AM-400" is not a noun. Thus, you can write "^1H NMR spectra were measured with (not on!) a Bruker AM-400 spectrometer."

[k] The subject of each experiment is given at the beginning; for synthetic procedures, the subject is simply the name of the product of the reaction. Indent (or tab), capitalize only the first letter of each word, and if necessary, use "wiggly underlining" to indicate bold-face typesetting. If the procedure you are following has been published previously, give a literature reference to the procedure you used.

[l] Note that certain unique (and amusing) customs have evolved with respect to English usage in describing experimental work. (a) The "I" form is almost never used; the passive or "we" form is preferred. (b) Be careful about tenses. Normally, an established fact (established by your work or by that of someone else) is given in the present tense, whereas a description of an experiment is given in the past tense (e.g., "Compound **1** is a high-melting solid", but "Compound **1** was treated with NaOH"). (c) A sentence should never be started with a numeral, an abbreviation, or a chemical formula. (d) A quantity of material is considered to be a "portion" and therefore is a singular and not a plural noun (e.g., "To the solution was added 25 mL of 1 *N* HCl"). (e) One should not omit "of" when describing a quantity (e.g., "The flask was charged with 20 mL of ether, 0.50 g of compound **34**, and 0.12 g of $LiAlH_4$"). (f) Capitalization at the beginning of a sentence is sometimes a problem. In general, Latin and Greek prefixes are not capitalized, so one would write "o-Chlorophenol was added. . ." and not "O-chlorophenol was added. . .".

[m] Note the ACS format for abbreviations. Some are used as lowercase (g, mg, sec, min, h), some as uppercase (NMR, GLC, IR, HPLC), and some as a mixture of lower- and uppercase (mL, μL). Periods are not used.

[n] Give the number of mols or mmols in parentheses after the weight. Although this is redundant, it provides a valuable check in case the all-important quantity is mis-typed. Also, note that the correct usage is mol, not mole, and mmol, not mmole.

[o] Be sure to note any unusual events (vigorous exotherms, formation of precipitates, copious evolutions of gas, color changes, etc.) that may occur in the course of a procedure.

addition was begun to minimize refluxing [p] the solvent. The mixture was stirred at room temperature under N_2 for 10 min and again cooled with a water bath as 17.5 mL (209 mmol) of pyrrolidine was added over 2 min. After 15 min the cooling bath was removed and the mixture was allowed to warm to room temperature overnight. The flask was immersed in an ice-water bath and 200 mL of 2 *M* HCl was added. [q] The mixture was transferred to a separatory funnel and the layers were separated. The aqueous layer was washed with CH_2Cl_2 (2 x 100 mL) [r] and the combined organic layers were washed with 100 mL [s] of a 4:1 mixture of saturated $NaHCO_3$ and brine. The aqueous layer was back-washed with 20 mL of CH_2Cl_2 and the combined organic layers were dried. The volatile material [t] was removed and the residue was distilled to afford 24.5 g (99%) [u] of amide 9 as a colorless oil, bp

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[p] Strictly speaking, "reflux" is a noun, not a verb, so one should not speak of "refluxing" of the solvent. For years, this has been a favorite object of Professors' red ink when correcting student reports. However, as Bill Bryson explains in his humorous book "The Mother Tongue: English & How It Got That Way," the language has been evolving since well before the time of Chaucer and it is still in a state of flux (or maybe reflux). It would seem that, in the case of "reflux", laboratory slang has carried the day and it is now so common to speak of "refluxing the solution" that we Professors must admit defeat and accept a new verb into our vocabulary.

[q] Note that it is the reaction flask that is immersed in the ice-water bath, not the reaction itself. That is, one should not refer to a "reaction" as if it were an object. Do not write, "the reaction was cooled to -78 °C. . ." , "the reaction was poured into water," or "HCl was added to the reaction."

[r] Note the correct form to use when describing extractions that involve multiple washing. Always indicate the quantity of solvent used for extractions.

[s] Give the quantity of wash solution used in extractive workups.

[t] Note that "volatile" is an adjective, not a noun. Therefore, it is not correct to say that "the volatiles were removed."

[u] The percent yield is given in parentheses after the quantity of reaction product. Yields are always based on the amount of limiting reagent employed at the start of the reaction, and should be calculated on a "percent of theoretical" basis. The stoichiometry of the reaction must therefore be considered in making a yield calculation. In this case, the limiting reagent was the 5-(phenylmethoxy)pentanoic acid (95 mmol), since both the N,N-carbonyldiimidazole and pyrrolidine were used in excess.

175-182 °C [v] (0.02 torr). [w] [x] IR: 1641 cm^{-1}. [y] ^1H NMR (250 MHz): [z] δ 1.59-2.00 (m, 8), 2.29 (br t, 2, J = 7), 3.37 (t, 2, J = 6.6), [aa] 3.45 (t, 2, J = 6), 3.50 (t, 2, J = 6), 4.49 (s, 2), 7.20-7.40 (m, 5). ^{13}C NMR (125 MHz): δ 21.62 (CH_2), [bb] 24.29 (CH_2), 26.00 (CH_2), 29.36 (CH_2), 34.33 (CH_2), 45.45 (CH_2), 46.45 (CH_2), 70.08 (CH_2), 72.78 (CH_2), 127.37 (CH), 127.52 (CH), 128.22 (CH), 138.50 (C), 171.35 (C). Anal. Calcd for $C_{16}H_{23}NO_2$: C, 73.53; H, 8.87; N, 5.36. Found: C, 73.27; H, 8.60; N, 5.23. [cc]

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[v] A quantity and its units are almost always separated by a space. Thus, you should write "24 g" instead of "24g" and "25 °C" instead of "25°C." The only common exception to this general rule is in degrees of rotation, in which case the quantity and the degree sign are not separated by a space (e.g., $\alpha = 34.5°$).

[w] In reporting boiling ranges, give the pressure. The unit "torr" (named after the Italian physicist Evangelista Torriceli) means, and is preferred to, "mm Hg."

[x] When a new compound is prepared, it should be isolated in pure form and adequate analytical data should be obtained to adequately characterize it with respect both to structure and degree of purity. This generally includes IR, ^1H NMR, ^{13}C NMR, and melting point (for solids), as well as other data that may be pertinent for a particular compound (e.g., optical rotation for chiral, enantiomerically-enriched compounds). For a compound not previously described in the literature, elemental analysis for at least C and H are required. If elemental analysis is not an appropriate criterion of purity (high-boiling, viscous oils, or in cases where only milligram quantities can be obtained), the purity and means of assessing it should be stated. If the method used to assess purity was spectral, you should provide (as an Appendix) photocopies of the spectra that were used.

[y] For most organic compounds, it is not necessary or desirable to list each and every infrared absorption band or mass spectrometric ion. Instead, only those that are readily characteristic of a functional group or structural feature are listed--in this case the amide carbonyl. This is because the ^1H NMR and ^{13}C NMR spectra give much more structural information. However, this is not the case for most inorganic or organometallic compounds, where NMR spectra are not as informative. In such cases, it is appropriate to list all of the IR bands, along with some indication of the intensity of the various bands. See Example 5.

[z] Note that the field strength is given, but not the solvent, since it was stated in the General Experimental Section that "unless otherwise noted, NMR spectra were measured as $CDCl_3$ solutions."

[aa] Note the form for reporting ^1H NMR data: chemical shift (multiplicity, number of hydrogens, coupling constants). The abbreviations used for multiplets are: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad. For higher multiplets, spell out the word; e.g., quintet, sextet, septet. For complex multiplets, use appropriate combinations; eg., dd, J = 4.5, 7.0 or dq, J = 2.3, 6.5. [Note that the order of the multiplet descriptors and coupling constants should match. That is, in the last example, the doublet coupling is 2.3 Hz and the quartet coupling is 6.5 Hz.]

[bb] Note the format for indicating carbon multiplicity when DEPT experiments have identified the methyls, methylenes, methines, and quaternaries.

[cc] Elemental analyses should be obtained for all new compounds. The accepted limits are ±0.4% for C, H, and N. Some journals require that authors submit actual photocopies of NMR spectra for all new compounds not characterized by elemental analysis. These spectra are sent to referees to use in judging if new compounds whose preparations are reported are of suitable purity.

Example 3. A Synthetic Procedure that gives Two Products

[1α(*R),2α,2(*E*)]- and [1α(*S**),2α,2(*E*)]-(±)-2-(4,8-Dimethyl-3,7-nonadienyl)-2-(hydroxymethyl)-β-[3-(phenylmethoxy)propyl]cyclopentanethanol (18a,b).** To 5.97 g (13.6 mmol) **[dd]** of crude lactones **17a,b** in a 1-L flask immersed in a cool tap water bath was added 140 mL of dry ether followed by 1.55 g (40.8 mmol) of solid $LiAlH_4$ **[ee]**, over 1 min. After the vigorous foaming had subsided the mixture was allowed to warm to room temperature. After 5 h, the mixture was cooled in an ice-water bath and 1.6 mL of water, 1.6 mL of 15% aqueous NaOH and 4.6 mL of water were added sequentially.[5] The cooling bath was removed, the mixture was allowed to warm to room temperature (20 min), and a white suspension resulted. Approximately 5 g of $MgSO_4$ was added and the mixture was filtered through a fine glass frit. The filtrate was concentrated first with a rotary evaporator **[ff]** and then at high vacuum for 12 h to afford 6.12 g of a colorless oil. Chromatography on 150 g of silica gel with 1:2 ethyl acetate/hexanes (1 L) followed by 1:1 ethyl acetate/hexanes (1 L) afforded 5.88 g (98%) of diols **18** as a colorless oil. Diols **18** were used as a 1:1 mixture of stereoisomers in subsequent reactions. Careful chromatography of a small ample of the diols on silica gel with 1:3 ethyl acetate/hexanes afforded the individual isomers **18a** and **18b**.

Less polar isomer: **[gg]** ^{1}H NMR (400 MHz): δ 1.18-1.32 (m, 2), 1.37-1.87 (m, 12), 1.60 (s, 6), 1.68 (d, 3, J = 0.7), 1.93-2.09 (m, 6), 2.15 (br s, 2), 3.38-3.66 (m, 6), 4.50 (s, 2), 5.09 (tt, 1, J = 1.4, 7.0), 5.14 (dt, 1, J = 1.1, 1.7), 7.20-7.40 (m, 5). ^{13}C NMR (100 MHz): δ 15.99 (CH_3), 17.67 (CH_3), 22.22 (CH_2), 23.34 (CH_2), 25.67 (CH_3), 26.05 (CH_2), 26.71 (CH_2), 27.03 (CH_2), 28.52 (CH_2), 35.03 (CH_2), 37.25 (CH_2), 39.693 (CH_2), 40.02 (CH), 48.14 (CH), 48.28 (C), 65.48 (CH_2), 65.84 (CH_2), 70.56 (CH_2), 72.93 (CH_2), 124.34 (CH), 124.90 (CH), 127.56 (CH), 127.69 (CH), 128.35 CH), 131.30 (C), 134.86 (C), 138.37 (C). Anal. Calcd for $C_{29}H_{46}O_3$: C, 78.68; H, 10.47.

[dd] Except in the case of highly precise, quantitative experiments, weights should generally be expressed to three significant figures. Note that the number of mmols cannot be given with a higher degree of precision than the weight. Thus, it is incorrect to say "To 5.97 g (13.621 mmol) of . . ."

[ee] Use chemical formulas for ordinary chemicals like $LiAlH_4$, NaOH, $MgSO_4$, $NaHCO_3$, etc. This saves a lot of page space in a journal.

[ff] Note that you remove solvent "with a rotary evaporator" and not "on a rotary evaporator", and you **never** "rotary evaporate," "rotovap", or "vap" something.

[gg] Sometimes a procedure, like this one, gives a mixture of products that are separated and individually characterized. In such cases, report the spectral and analytical data as separate, indented paragraphs immediately following the description of the experimental procedure and workup. In this case, it was not determined which diastereomer had which of the two possible structures, so they are described simply as the "less polar" and "more polar" isomers. If their structures had been assigned, then these data paragraphs would have been entitled "**Compound 18a**" and "**Compound 18b**" (or vice versa).

Found: C, 78.39; H, 10.59.

More polar isomer: ^{1}H NMR (400 MHz,): δ 1.14 (br ddd, 1, J = 6, 11, 13), 1.60 (s, 6), 1.68 (s, 3), 1.32-1.85 (m, 13), 1.87-2.10 (m, 6), 2.88 (br s, 2), 3.43 (d, 1, J = 11.8), 3.46 (t, 2, J = 6.6), 3.53 (dd, 1, J = 5.0, 10.5), 3.55 (d, 1, J = 11.8), 3.66 (dd, 1, J = 7.0, 10.5), 4.50 (s, 2), 5.09 (tt, 1, J = 1.4, 7.0), 5.12 (dt, 1, J = 1.4, 7.0), 7.23-7.42 (m, 5). ^{13}C NMR (100 MHz): δ 15.99 (CH_3), 17.68 (CH_3), 22.11 (CH_2), 23.49 (CH_2), 25.68 (CH_3), 26.70 (CH_2), 27.17 (CH_2), 27.38 (CH_2), 30.28 (CH_2), 34.35 (CH_2), 37.60 (CH_2), 37.96 (CH), 39.70 (CH_2), 48.02 (C), 50.55 (CH), 64.93 (CH_2), 65.95 (CH_2), 70.67 (CH_2), 72.98 (CH_2), 124.34 (CH), 124.89 (CH), 127.54 (CH), 127.68 (CH), 128.35 (CH), 131.31 (C), 134.83 (C), 138.43 (C). Anal. Calcd for $C_{29}H_{46}O_3$: C, 78.68; H, 10.47. Found: C, 78.47; H, 10.44.

Example 4. Preparation of a Previously-Reported Compound

2-Oxocyclopentaneacetic acid (4). **[hh]** A 3-L three-neck, round-bottom flask **[ii]** **[jj]** was flushed with dry N_2 and charged with 50.8 g (1.06 mol) of NaH (50% oil dispersion). The NaH was washed three times with hexane, the supernatant being carefully decanted each time under a generous flow of N_2, and suspended in 1 L of THF. The reaction vessel was fitted with a reflux condenser, mechanical stirrer, and a 250 mL pressure-equalizing dropping funnel containing 129 mL (1.04 mol) of methyl 2-oxocyclopentanecarboxylate and 120 mL of THF. Positive N_2 flow was maintained during the apparatus manipulation. The keto ester solution was slowly added to the reaction vessel with stirring. After the addition was complete, the mixture had reached a consistency that allowed for magnetic stirring, if desired. The mixture was heated at reflux **[kk]** for 2 h, then cooled to room temperature. A 250-mL **[ll]** addition funnel containing 122 mL (1.10 mol) of ethyl bromoacetate (technical grade, 96%) and 130 mL of THF was attached to the flask, and the halide solution was added to the mixture at a rate sufficient to maintain gentle reflux. After the addition was complete, the mixture was heated at reflux overnight. After cooling to room temperature, 750 mL of water was added to the flask and the contents were transferred to a separatory funnel. The layers were separated and the aqueous phase was extracted with ethyl acetate (2 x 100 mL). The combined organic layers were dried and evaporated. **[mm]** Distillation of the brown, oily residue

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[hh] If the procedure you used is essentially the same as has already been published in the literature, give a reference here. In such a case, you need not describe your procedure at all unless you have modified the one that is published in the literature. Regardless, if you have obtained better spectral data than has previously been published, you should report it. In this case, although the compound has been reported in the literature, the procedure used to prepare it is not the same as the published procedure. Therefore, the entire procedure must be given.

[ii] Generally speaking, journal editors do not like authors to use precious page space describing routine apparatus. That is, if every preparative procedure started out "A flame-dried, 100-mL, three-necked, round-bottomed flask equipped with a magnetic stirring bar, nitrogen inlet, and reflux condenser was charged with . . .", we would waste many pages of journal space and perhaps prevent someone's useful paper from being published. In this case, the size and nature of the reaction flask is stated because the reaction is one that is prone to evolve copious amounts of hydrogen from the reaction of the NaH and the β-keto ester (1.04 mols--a total of over 22,000 mL of hydrogen!) and it is wise to alert the potential user that a lot of space needs to be provided for ebullition.

[jj] Note that "round-bottom" is an adjective, not a noun. Thus, you can say that "a 100-mL round-bottom flask was dried. . ." but not that "a 100-mL round-bottom was dried. . ."

[kk] [This is the way we Professors really like it!]

[ll] Note that compound adjectives and adverbs are hyphenated; e.g., "50 mL of ether was added from a 100-mL dropping funnel."

[mm] Note that it is not necessary to specify that the drying agent was $MgSO_4$ or that the evaporation was carried out with a rotary evaporator at reduced pressure because this information was given in the General Experimental Section.

through a standard Claisen head gave 180 g (76%) of a colorless the keto diester as an oil, bp 95 °C (0.05 torr). IR: 1745 (shoulder), 1730, 1200 cm^{-1}. ^1H NMR (250 MHz): δ 1.20 (t, 3, J = 7), 1.8-2.65 (m, 6), 2.60 (d, 1, J = 18), 2.29 (d, 1, J =18), 3.63 (s, 3), 4.03 (q, 2, J = 7). ^{13}C NMR (50 MHz): δ 13.5, 19.0, 32.8, 36.9, 37.8, 52.0, 57.0, 60.1, 170.1, 170.2, 212.8.

A solution of the foregoing diester (25.15 g, 0.653 mol) in 300 mL of conc HCl was heated at reflux for 6 h. After cooling, most of the water and HCl were removed by distillation at 10-15 torr through a standard Claisen head. The residual water (40-50 mL) was removed by azeotropic separation with benzene (Dean-Stark trap). The benzene solution was concentrated to a brown residue that was distilled to afford 74.4 g (80%) of a slightly yellow oil, bp 117 °C (0.04 torr). The oil was transferred to a 250-mL beaker where slow crystallization took place. The hard crystalline mass was broken up, ground with a mortar and pestle, and dried in a vacuum dessicator over P_2O_5. Keto acid **4** obtained in this way had mp 42-50 °C and could be recrystallized from ether-hexane to give a white powder, mp 50-51 °C (lit. 51 °C).[6] **[nn]** IR ($CHCl_3$): 1740, 1710 cm^{-1}. ^1H NMR (250 MHz): δ 1.3-2.9 (m, 9), 11.3 (s, 1). ^{13}C NMR (50 MHz): δ 19.9, 28.5, 33.0, 36.6, 44.8, 176.0, 219.3. **[oo] [pp]**

[nn] When citing literature values for physical properties, always give an appropriate reference.

[oo] Because IR and NMR spectral data have not previously been reported for this compound, these data are given here.

[pp] Because the compound was identified by comparison of its melting point with the published one, it is not necessary to report elemental analysis.

Example 5. A Procedure that Is Carried Out With the Use of a Dry Box; NMR Data Given In Tabular Form

$(PMe_3)_4Ru(\eta 2\text{-}C_6H_4)$ (1). In a recycling inert atmosphere box (N_2), a 250-mL flask equipped with a side arm was charged with 1.50 g (3.15 mmol) of $Ru(PMe_3)_4(Cl)_2$ and 100 mL of diethyl ether and a magnetic stirring bar. Into a Schlenk tube was placed 2.31 mL (2.2 equiv., 6.9 mmol) of a 3.0 *M* solution of phenylmagnesium bromide in ether, along with 5 mL of additional ether. Both vessels were brought out of the drybox, carefully degassed, and the N_2 atmosphere was replaced with argon (it is essential that all solutions be N_2-free because $Ru(PMe_3)_4(Ph)_2$ forms a complex with N_2). The Grignard reagent was added by cannula to the ether slurry of $Ru(PMe_3)_4(Cl)_2$ at room temperature over a 5- to 10-minute period. The solution was then stirred under argon for approximately 3 h at room temperature, until no orange, solid $Ru(PMe_3)_4(Cl)_2$ remained. The resulting solution, including magnesium salts, was transferred by cannula to a glass reaction vessel fused to a Kontes vacuum adaptor. The vessel was partially degassed. The vessel was then closed and heated to 85 °C for 8 h. The solution turned dark brown upon heating; the reaction is complete when the solution becomes light brown in color while at 85 °C. After this time, the solvent was removed under reduced pressure, and the residue was extracted 3 times with a total of 150 mL of pentane, stirring for 5 min after each addition of solvent. Removal of the pentane in vacuo left a pale-yellow or white solid in 55-70% yield. This material is pure enough (ca. 95%) for synthetic purposes. Alternatively, crystallization from pentane provided analytically-pure samples of 1 in 35-50% yield. IR (Nujol): **[qq]** 2055 (m), 1974 (m), 1931 (m), 1886 (s), 1819 (m), 1788 (m), 1767 (m), 1705 (m), 1570 (s), 1523 (m), 1412 (s), 1402 (s), 1295 (s), 1277 (s), 1188 (m), 1140 (m), 1115 (m), 1070 (m), 1028 (s), 1000 (m), 932 (s), 885 (s), 780 (m), 705 (s), 674 (s), 660 (s), 610(s). MS (EI): **[rr]** *m/z* 482 (M^+). Anal. Calcd. for $C_{18}H_{40}P_4Ru$: C, 44.90; H, 8.37. Found: C, 45.17; H, 8.38. **[ss]**

[qq] Note that the medium used for the infrared spectrum used is specified since it differs from that stated in the General Experimental Section. For this organometallic compound, the infrared spectrum is a principal method of characterization, so all of the resonances are listed, along with parenthetical descriptors; s = strong, m = medium, w = weak.

[rr] If it was not given in the General Experimental Section, state the mass spectral mode; EI = electron impact, CI = chemical ionization, FAB = fast atom bombardment. Note that the units for mass spectral data are "*m/z*," not "*m/e*" as was formerly the case.

[ss] In some cases, such as this preparation of an organometallic compound and the subsequent kinetic analysis of its thermolysis, it is desirable to report the NMR data in tabular, rather than narrative form. The NMR data for this molecule, and some related complexes, are given in the attached table.

Example 6. Description of a Kinetic Analysis

Kinetic evaluation of the thermolysis of $Ru(PMe_3)_4(Ph)(Me)$ in C_6D_6. Into a 5.00-mL volumetric flask was weighed 35.6 mg (0.0716 mmol) of $Ru(PMe_3)_4(Ph)(Me)$ and 28 mg of ferrocene as an internal standard. Enough C_6D_6 was added to the flask to make a 0.0143 *M* solution. In a typical experiment, 0.700 mL of this solution was added by syringe to a thin-walled, 9-inch NMR tube. The tube was degassed on a vacuum line, the appropriate amount of PMe_3 was added by vacuum transfer and the tube was flame sealed to give a tube length of 8.5 inch. The tubes were heated at 110 °C in a factory-calibrated Neslab Exocal Model 251 constant temperature bath filled with Dow Corning 200 silicone Fluid. Individual tubes were removed from the bath at different times for analysis; each was frozen rapidly in ice water immediately after removal from the bath. All reactions were monitored to greater than 3 half-lives by ambient-temperature ^1H NMR spectrometry by integrating the PMe_3 resonance of the starting material at δ 0.99 ppm vs. the ferrocene internal standard. To obtain accurate integrations, the spectra were taken with a single acquisition and double checked with a second acquisition after a delay of at least 10 T_1. All kinetic plots displayed excellent linearity with correlation coefficients of 0.98 or better. A plot of k_{obs} vs. $l/[L]$ is shown in Figure 4 and displays a correlation coefficient of 0.99.

Table 13. ^{1}H NMR Spectroscopic Data[a] [tt]

δ (ppm)	Multiplicity	J (Hz)	Integral	Assignment
$(PMe_3)_4Ru(\eta^2\text{-}C_6H_4)$ (1)				
0.78	t	4.4	18	trans-PMe_3
1.28	d	2.6	18	cis-PMe_3
7.26	br s		2	aromatic
7.31	br s		2	aromatic
$(PMe_3)_4Ru(\eta^2\text{-}C_6D_4)$ (**1-d$_4$**)[b]				
0.78	t	4.4	18	trans-PMe_3
1.28	d	2.6	18	cis-PMe_3
$(PMe_3)_4Ru(Me)(Ph)$ (2)[b]				
-0.033	dq	7.7, 3.5	3	Ru-Me
0.96	t	5.2	18	trans-PMe_3
1.04	d	5.0	9	cis-PMe_3
1.22	d	4.7	9	cis-PMe_3
7.13	m		1	aromatic
7.19	m		2	aromatic
7.88	br s		2	aromatic
$(PMe_3)_4Ru(\eta^2\text{-}OC(CH_2)C_6H_4)$ (4)[c]				
1.02	t	6.0	18	trans-PMe_3
1.42	d	6.9	9	cis-PMe_3
1.45	d	6.0	9	cis-PMe_3
3.39	d	1.2	1	$OC(CH_2)Ar$
3.95	br s		1	$OC(CH_2)Ar$
6.61	tq	7.3, 1.0	1	aromatic
6.63	t	7.1	1	aromatic
7.11	dt	7.6, 1.6	1	aromatic
7.46	m		1	aromatic

a) The multiplicities d and t, when applied to the PMe_3 resonances, refer to apparent splitting patterns. Accordingly, the values reported for J do not reflect true coupling constants. b) C_6D_6, 20 ° C. c) THF-d_8, -20 °C.

[tt] Some journal editors (the Editor of the *Journal of Organic Chemistry*, for example) frown on representation of spectral data in tables, because this format is very wasteful of page space. For example, compare the above list with the following one: $(PMe_3)_4Ru(\eta^2\text{-}C_6H_4)$ (**1**): δ 0.78 (t, 18, J = 4.4, trans-PMe_3), 1.28 (d, 18, J = 2.6, cis-PMe_3), 7.26, (br s, 2), 7.31 (br s, 2). $(PMe_3)_4Ru(\eta^2\text{-}C_6D_4)$ (**1-d$_4$**): δ 0.78 (t, 18, J = 4.4, trans-PMe_3, 1.28 (d, 18, J = 2.6, cis-PMe_3. $(PMe_3)_4Ru(Me)(Ph)$ (2): δ -0.033 (dq, 3, J = 7.7, 3.5, Ru-Me), 0.96 (t, 18, J = 5.2, trans- PMe_3), 1.04 (d, 9, J = 5.09, cis-PMe_3), 1.22 (d,9, J = 4.7, cis-PMe_3), 7.13 (m, 1), 7.19 (m, 2), 7.88 (br s, 2). $(PMe_3)_4Ru(\eta^2\text{-}OC(CH_2)Ar)$ (**4**): δ 1.02 (t, 18, J = 6.0, trans-PMe_3), 1.42 (d, 9 , J = 6.9, cis-PMe_3), 1.45 (d, 9, J = 6.0, cis-PMe_3), 3 39 (d, 1, J = 1.2, $OC(CH_2)Ar$), 3.95 (br s, 2, $OC(CH_2)C_6H_4$), 6.61 (tq, 1, J = 7.3, 1.0), 6.63 (t, J =7.1), 7.11 (dt, J = 7.6, 1.6), 7.46 (m).

On the other hand, some journals, *Organometallics* being one, actually recommend that authors use the more easily readable tabular format. In writing your reports, you should consult with your research director about where your work is most likely to be published. In addition, it is sometimes desirable to use tabular format if it permits comparison of chemical shifts and coupling constants of similar nuclei in a series of related compounds and if such comparison is an important part of the discussion of structure assignment.

Example 6. References [uu]

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[uu] Reference format varies from journal to journal. The format given here, and that expected in Berkeley Department of Chemistry First Year Reports, is the ACS journal format. Carefully note the format, including punctuation. Note that author's names are given in the order Lastname, Initials; and that the semicolon is omitted after the initials of the last author. Journal name is italicized (underlined if not using a word processor), publication year is bold-face ("wiggly" underlined if not using a word processor), volume number is italicized, followed by the number of the first page of the article and a period.