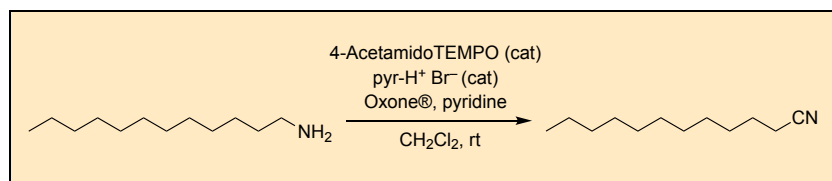# Catalytic, Metal-Free Oxidation of Primary Amines to Nitriles

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*Checked by Thomas R. DeVino, Andreas R. Rötheli, and Margaret Faul*



## Procedure (Note 1)

A. *Dodecanenitrile*. A 1-L, three-necked, round-bottomed flask is equipped with an oval Teflon-coated magnetic stir bar, a rubber septum, a nitrogen line fitted to a glass bubbler filled with mineral oil, and a 250-mL pressure-equalizing addition funnel capped with a rubber septum pierced with a needle (Notes 2, 3, 4, and 5) (Figure 1).

While under a continuous flow of nitrogen, the flask is dried with a heat gun and then allowed to cool to room temperature. The needle on the addition funnel is removed. Then the septum on the flask is removed and the flask is charged with 200 mL of dichloromethane (Note 6), Oxone (80.0 g, 130 mmol, 2.6 equiv) (Note 7), and pyridinium bromide (365 mg, 2.25 mmol, 0.045 equiv) (Note 8) (Figure 2). The heterogeneous mixture is stirred for 15 min until a faint yellow color develops (Figure 3).

Dry pyridine (20 mL, 19.6 g, 250 mmol, 5 equiv) (Note 9) is then added *via* syringe through the rubber septum (Figure 3), upon which the yellow color of the reaction mixture intensifies (Figure 4). The reaction mixture is
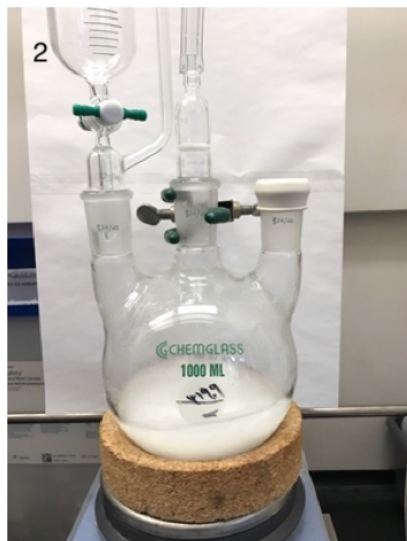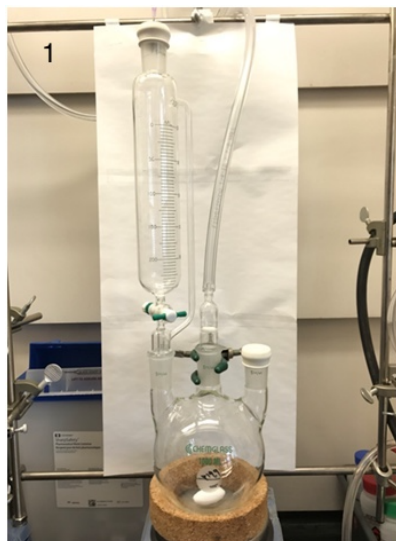
**Figure 1. Initial reaction flask setup for drying (provided by checker);**
**Figure 2.** **Initial mixture of Oxone and pyridinium bromide in dichloromethane (provided by checker)**
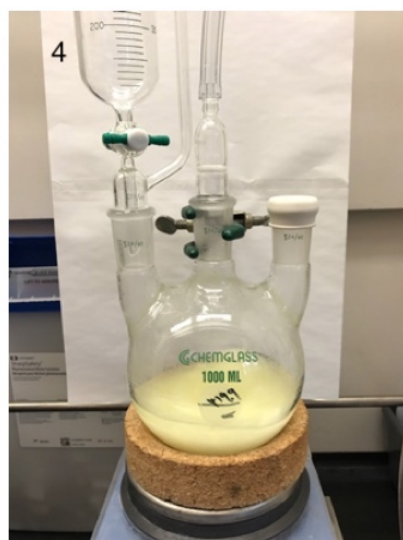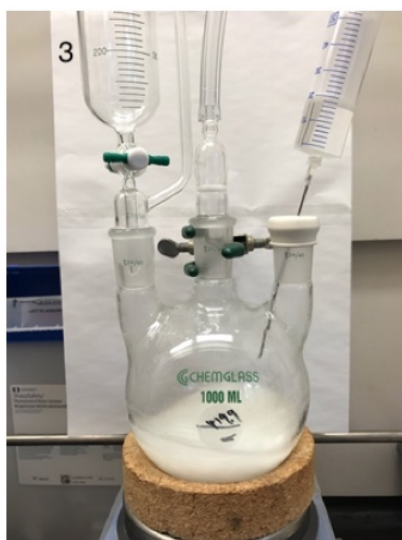


**Figure 3. Reaction mixture prior to the addition of pyridine (provided by checker); Figure 4. Reaction mixture after the addition of pyridine (provided by checker)**

stirred for 1–2 min, the septum is then removed and the 4-acetamidoTEMPO catalyst (533 mg, 2.50 mmol, 0.050 equiv) (Note 10) is added in one portion. The septum is placed back on the flask and the reaction mixture, which darkens upon addition of the catalyst (Figure 5), is stirred for 15–30 min.
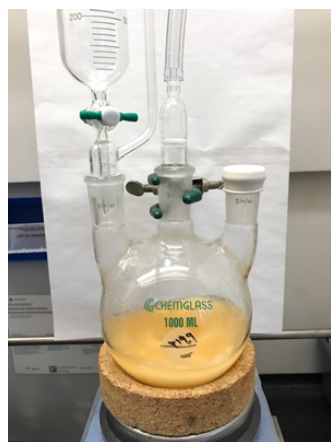


**Figure 5. Reaction mixture after the addition of catalyst
(provided by checker)**

A clean, dry 125-mL Erlenmeyer flask is charged with dodecylamine (9.24 g, 49.9 mmol, 1 equiv) (Note 11) and 100 mL of dichloromethane. The Erlenmeyer flask is swirled to dissolve the amine. The septum on the addition funnel is removed and the homogenous solution of the amine is transferred to the addition funnel through the use of a funnel. The Erlenmeyer flask is then rinsed with additional dichloromethane (10 mL) to ensure complete transfer and to rinse any residual amine from the funnel. The funnel is then removed and the septum is placed back onto the addition funnel. An additional nitrogen line is added through the septum of the addition funnel and the addition rate is adjusted to ensure a constant delivery of the amine solution at approximately 40–50 mL/h (Figure 6). Addition of the amine takes approximately 2–2.5 h to complete, at which point additional dichloromethane (20 mL) is added *via* syringe to the funnel (Note 12) (Figure 8) allowing for any residual white solid (Figure 7) to be rinsed into the reaction flask. The light yellow reaction mixture (Figure 8) is stirred at 23 °C (Note 13) for an additional 9–15 h (Note 14).

**Figure 6. Reaction flask setup for the addition of the amine solution in dichloromethane (provided by checker); Figure 7. Residual solids in the addition funnel after amine addition (provided by checker)**
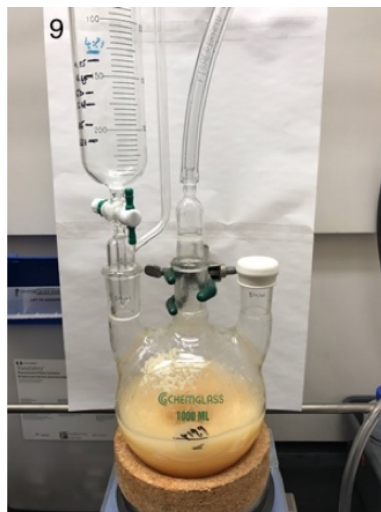



**Figure 8. Rinse of residual solid with dichloromethane using a syringe (provided by checker); Figure 9. Reaction flask with final reaction mixture before extended stirring. (provided by checker)**

During this time the reaction mixture turns from a yellow suspension (Figure 9) to a clear yellow solution with sticky yellow solids along the wall of the round-bottomed flask (Figure 10)



**Figure 10. Reaction flask with final reaction mixture after extended stirring (provided by checker)**

A 600–mL medium porosity, sintered glass Büchner funnel is filled with 150 g of silica gel (Note 15), and a small filter paper (90 mm diameter, coarse porosity) is placed on top of the silica gel. The funnel is then fitted to a 1–L round-bottomed flask, set up for vacuum filtration, and the reaction mixture is poured through the filtration setup (Figure 11). The reaction flask is rinsed with an additional 300 mL of dichloromethane and poured through the filtration setup (Note 16) (Figure 12). The yellow solids remain in the original flask (Figure 13).
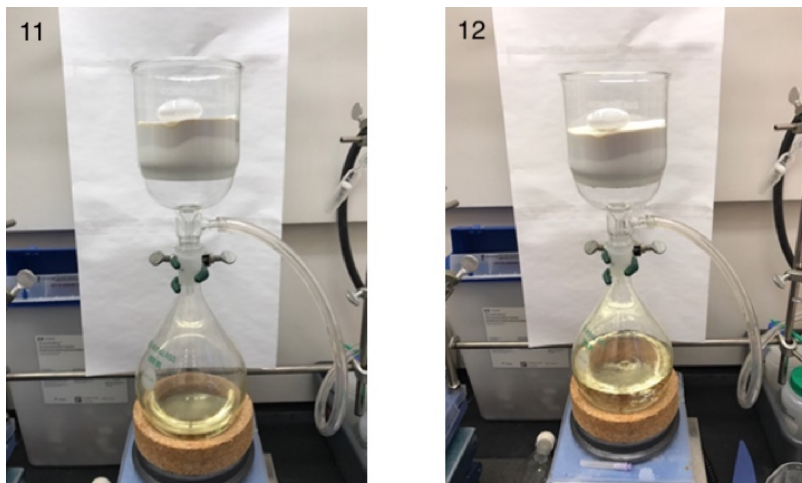
**Figure 11. Filtration of reaction mixture through silica gel. (provided by checker); Figure 12. Filtration setup after dichloromethane wash (provided by checker)**



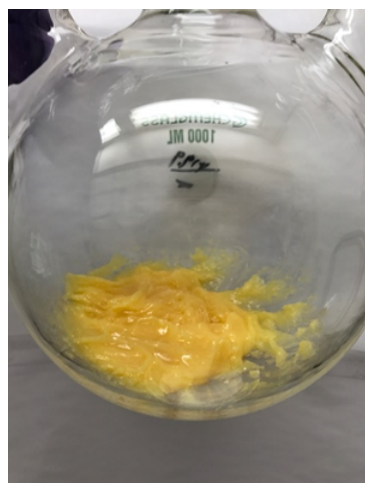**Figure 13. Residual solids in the reaction flask after the dichloromethane wash (provided by checker)**

The filtrate is concentrated by rotary evaporation (30–-40 °C, 500 mmHg then to approx. 15 mmHg for 15–30 min) (Note 17) to afford dodecanenitrile (8.80 g, 94%) (Notes 18 and 19) as a pale yellow oil (Notes 20, 21, and 22). However, the title compound is reported in the literature as a clear,

colorless oil.[3] Colorless product is obtained by transferring the pale yellow crude oil (Note 23) to a 25-mL round-bottomed flask from the 1-L round-bottomed flask. The remaining oil in the 1-L round-bottomed flask is rinsed with dichloromethane (2 x 5 mL) and transferred to the 25-mL round-bottomed flask to ensure all the oil is transferred. Then the dichloromethane was removed by rotary evaporation (30–40 °C, 500 mmHg then to approx. 15 mmHg for 15–30 min). The 25-mL round-bottomed flask is then set up for bulb-to-bulb distillation (Kugelrohr, bath temp: 200 °C, 10–20 mmHg) (Figure 14) to afford analytically pure dodecanenitrile (8.37 g, 93% yield) (Notes 24, 25, 26, and 27), as a clear colorless oil.
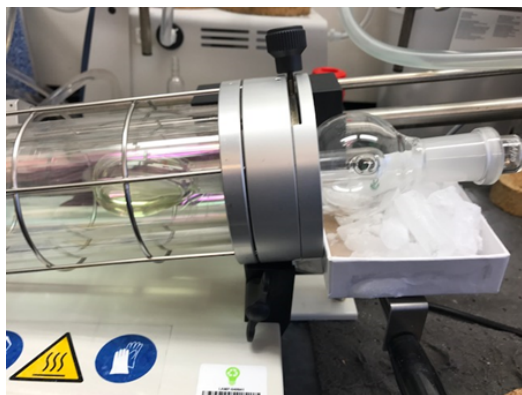


**Figure 14. Kugelrohr setup at the beginning of the distillation (provided by checker)**

## Notes

1. Prior to performing each reaction, a thorough hazard analysis and risk assessment should be carried out with regard to each chemical substance and experimental operation on the scale planned and in the context of the laboratory where the procedures will be carried out. Guidelines for carrying out risk assessments and for analyzing the hazards associated with chemicals can be found in references such as Chapter 4 of "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at https://www.nap.edu/catalog/12654/prudent-practices-in-the-laboratory-handling-and-management-of-chemical).

See also "Identifying and Evaluating Hazards in Research Laboratories" (American Chemical Society, 2015) which is available via the associated website "Hazard Assessment in Research Laboratories" at https://www.acs.org/content/acs/en/about/governance/committees/chemicalsafety/hazard-assessment.html. In the case of this procedure, the risk assessment should include (but not necessarily be limited to) an evaluation of the potential hazards associated with dichloromethane, Oxone, pyridinium bromide, pyridine, 4-acetamidoTEMPO, dodecylamine, and silica gel, as well as the proper procedures for using a Kugelrohr apparatus.

2. The submitters used a 1-L one-necked, round-bottomed flask equipped with an oval Teflon-coated magnetic stir bar (40 mm x 20 mm) and capped with a septum that is connected *via* a nitrogen line inlet and an outlet fitted to a glass bubbler filled with mineral oil (Figure 15). The submitters flame dried the flask prior to setting up the reaction.
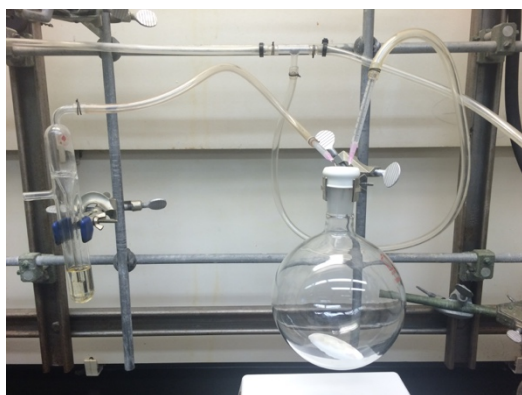


**Figure 15. Initial reaction flask setup for flame drying (provided by submitter)**

3. The submitters note that nitrogen gas is sufficient to establish an anhydrous atmosphere, and is less expensive than argon. However, if argon is available, it should be used as it results in slightly higher isolated yields ~2% greater than those obtained when nitrogen is used. The isolated yields reported in this manuscript were obtained when nitrogen gas was used.

4. A 250-mL constant-rate addition funnel was used, but a 125-mL funnel would suffice for this scale. Due to the use of a 1-L one-necked, round-

bottomed flask the submitters had to mount a 500-mL constant-rate addition funnel available from Kontes (cat. # 186-634620-0500) after the addition of the 4-acetamidoTEMPO catalyst (Figure 16). Prior to fitting to the reaction flask, the funnel was calibrated with dichloromethane to allow for the addition of 40–50 mL of solution per hour (~ 30 drops per minute) and the graduated hash mark where the rod was located was noted.  A less sophisticated pressure-equalizing addition funnel would likely suffice as long as the rate of addition does not exceed 40–50 mL/h.
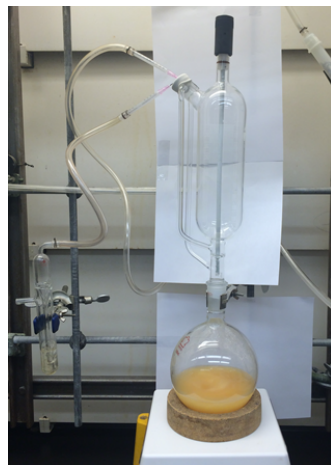


**Figure 16.  Reaction Flask Fitted with the Pressure-Equalizing Addition Funnel (provided by submitter)**

5.   The submitters report that the failure to purge the headspace of the addition funnel with nitrogen or argon gas results in the build-up of a white crystalline solid during the course of the addition of the amine substrate. This solid is likely the result of the reaction of the amine substrate with residual carbon dioxide to form a carbamic acid.[8] Build-up of the white solid can result in a slowed and problematic addition rate. However, the formation of the solid does occur to some extent even under the best attempts to remove the carbon dioxide, but its formation does not influence isolated yields of nitrile as the carbamic acid is in equilibrium with the free amine under the reaction conditions and the oxidation proceeds.

6. Dichloromethane (ACS reagent grade ≥ 99.5%, <0.01% water, 40–150 ppm amylene stabilized) was purchased from Sigma-Aldrich and was used as received. The submitters report that this solvent contained the appropriate quantity of water to solubilize a small portion of the oxidant. The reaction fails to proceed under strictly anhydrous conditions[4] and significant quantities of aldehyde byproduct result when the water content is too high. For these reasons all other sources of water should be avoided.

7. Oxone monopersulfate was purchased as a granular white powder from Alfa Aesar and used as received. Due to the hygroscopic nature of Oxone, a fresh bottle is recommended, and it should be stored in a tightly closed container in a dry location such as a desiccator. The submitters reported that additional moisture absorbed by the Oxone has a detrimental effect on the oxidation, and due to instability at high temperatures drying of the reagent is not recommended. The Oxone should be added to a rapidly stirred solvent to avoid difficulties in initiating the stirring. Oxone is a commercially available triple salt of empirical formula 2 $KHSO_5 \bullet KHSO_4 \bullet K_2SO_4$, and has a combined molecular mass of 614.7 g/mol. Each molar quantity of the triple salt contains 2 moles of the terminal oxidant ($KHSO_5$), which has a molecular mass of 152.2 g/mol. The oxidation requires at least 2.0 equivalents of $KHSO_5$ to proceed; however, it is beneficial to use an excess as it simplifies the work-up process (Note 12). The amount of Oxone employed in this procedure is 130 mmol (80.0 g, 2.6 equiv) per 50 mmol of amine substrate, and is arrived at by accounting for the fact that commercial Oxone contains only 42–44 % by mass of the active component ($KHSO_5$).[5] As a result, 33.6 g (220 mmol, 4.4 equiv relative to the starting amine) of the terminal oxidant ($KHSO_5$) is present in the reaction mixture. This was calculated as follows:

   50 mmol amine x 4.4 equiv. = 220 mmol oxidant
   0.220 mol $KHSO_5$ x (152.2 g $KHSO_5$/1 mol $KHSO_5$) = 33.5 g $KHSO_5$
   33.5 g $KHSO_5$/0.42 = 80.0 g Oxone

8. Pyridine hydrobromide (98%) was purchased from Sigma Aldrich and used as received. Pyridinium bromide is very hygroscopic and must be kept dry and moisture-free. The submitters report that it is essential that high-quality pyridinium bromide, that does not contain any tribromide as the counterion, is used. Alternatively, a high-quality sample of

**69**

pyridinium bromide may be obtained from the reaction of freshly distilled *tert*-butyl bromide with dry pyridine.[6]

9. Pyridine (ACS reagent grade), was purchased from Sigma Aldrich and used as received. Submitters purchased from J.T. Baker and dried by magnetically stirring with calcium hydride for 24 h at room temperature in an oven-dried flask capped with a septum, then distilled under nitrogen collecting the fraction boiling at 114–116 °C into a flame-dried flask. While the oxidation requires at least 4 equivalents of pyridine to proceed,[4] the additional equivalent of pyridine ensures the amine substrate does not act as the base. When the amine substrate is protonated it fails to undergo any oxidative processes.

10. 4-AcetamidoTEMPO (4-Acetamido-2,2,6,6-tetramethylpiperidine 1-oxyl) is commercially available in 98+% from Alfa Aesar. However, this nitroxide is easily and inexpensively prepared in multi-molar quantities from known procedures.[7] The submitters report that the catalyst should be dried in an Abderhalden drying pistol at 56 °C (acetone, 10 mmHg) overnight prior to use; mp 144–146 °C (lit.[7] 145–147 °C). The checkers used commercial 4-acetamidoTEMPO as received, without any further drying.

11. Dodecylamine (98% purity) was purchased from Sigma-Aldrich and used as received. The amine turns to a light yellow color over time, but is easily purified by bulb-to-bulb distillation (Bath temp: 155 °C, 15 mmHg), if necessary prior to use. Dodecylamine was used as received by the checkers.

12. A small quantity of white solid forms upon the wall of the addition funnel (Note 4) and the additional 20 mL of dichloromethane should be added using a syringe to rinse the walls of the addition funnel so as to remove as much white solid as possible. The submitters removed the rubber septum and used a glass pipette to rinse the addition funnel (Figure 17)
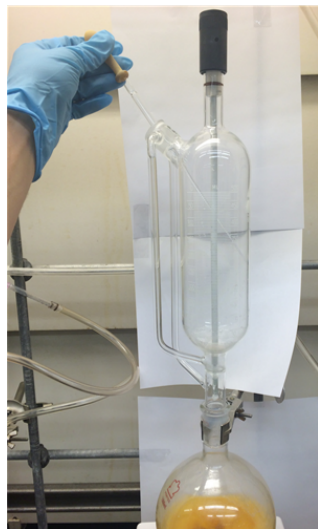
**Figure 17. Residual solid is rinsed with dichloromethane with a long glass pipette (provided by submitter)**

13. The heterogeneous mixture may become difficult to stir and the stir bar may stop after the addition of the amine is complete. This does not seem to affect the yield as the oxidation is complete at this time. Additionally, the reaction temperature may increase slightly during the amine addition to approximately 30 °C.

14. The oxidation of the amine substrate to the corresponding nitrile is quite rapid and is completed usually within 1 h of addition. However, due to the requirement to use an excess of an external base, pyridine, working the reaction up at this time would require an extractive work-up with acid. Removal of the excess pyridine and isolation of the product is greatly complicated, and not feasible for acid-sensitive substrates. Thus, the reaction is allowed to stir for the given period of time as the excess oxone efficiently oxidizes the majority of the excess pyridine to pyridine-*N*-oxide.[4] The pyridine-*N*-oxide is easily removed upon work-up.

15. Silica gel was purchased from Acros Organics and the particle size was 0–60 μm (60 Å). The submitters used silica gel from Silicyle or ZEOChem and the particle size was 40–63 μm (230–400 mesh).

16. The submitters report that the filtration should be stopped when the colored bands (Figure 18) have moved to within 1 cm of the glass frit.

The light yellow band is associated with the small excess of pyridine present and should not be collected. On occasion a light purple band is visible and should not be collected either. The checkers did not observe these bands right after filtration. However, they did appear upon aging of the filter cake under ambient conditions.
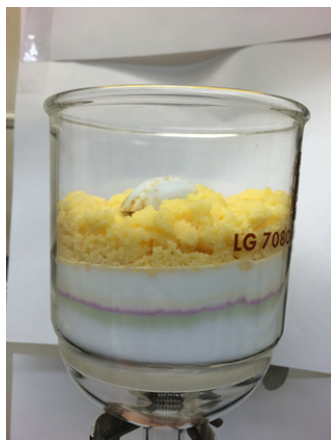


**Figure 18. Colored bands observed during filtration**
**(provided by submitter)**

17. The submitters report that placing a small wooden boiling stick into the flask facilitates the removal of excess dichloromethane while under vacuum, and can easily be removed prior to obtaining a final weight of compound without significantly altering the isolated yield.
18. Corresponds to weight adjusted yield based on purity determined by quantitative ^{1}H NMR analysis (see below for ^{1}H QNMR spectrum)
19. The submitters found that the refractive index of the crude product was $n_D^{20} = 1.4353$, and was in agreement with the literature value of $n_D^{20} = 1.4361$.[2]
20. Additional runs of the procedure by the Checkers afforded the title compound with yields of 88% and 92%.
21. The submitters report that after storage over a period of 6 months in a refrigerator at 6 °C, in a glass vial capped with a septum, the compound maintains its initial light yellow color with no apparent decomposition. A check of the refractive index was done $n_D^{20} = 1.4368$, which is consistent with the value reported in the literature.[2]

22. The procedure described will afford the title compound with purity sufficient for most purposes. Quantitative $^1$H NMR analysis showed this product was of 97% purity (18.9 mg of analyte with 21.2 mg benzyl benzoate of 100% purity purchased from Sigma-Aldrich as a standard; see below for $^1$H QNMR spectrum) The submitters used dimethylfumarate as an internal standard and also assessed purity by elemental analysis. calcd for $C_{12}H_{23}N$: C 79.49, H 12.79, N 7.72, found C 79.22, H 12.97, N 7.90.

23. A small portion (*i.e.* 18.9 mg) was removed for quantitative $^1$H NMR analysis.

24. The distillation removes any trace impurities and results in a clear, colorless oil. Quantitative $^1$H NMR analyses showed that the distilled products were of >99.5% average purity. (*e.g.* 18.6 mg of analyte with 21.4 mg benzyl benzoate of 100% purity purchased from Sigma-Aldrich as a standard; see below for $^1$H QNMR spectrum). Note: The submitters used dimethylfumarate as an internal standard.

25. The submitters found that the refractive index of the distilled product was $n_D^{20}$ = 1.4364 and was in agreement with the literature value of $n_D^{20}$ = 1.4361.[2]

26. The product was characterized as follows:[9] $^1$H NMR (500 MHz, CDCl$_3$) δ: 0.88 (t, *J* = 6.9 Hz, 3H), 1.28 (m, 14H), 1.44 (quin, *J* = 7.3 Hz, 2H), 1.65 (quin, *J* = 7.4 Hz, 2H), 2.33 (t, *J* = 7.2 Hz, 2H). $^{13}$C NMR (125 MHz, CDCl$_3$) δ: 14.3, 17.3, 22.9, 25.6, 29.0, 29.5, 29.7, 32.1, 120.0. FT–IR (neat, ATR): 2923, 2854, 2246, 1666, 1645, 1466, 1427, 1378, 1364, 1329, 1246, 1163, 1122, 1070, 1054, 931, 907, 882, 846, 818, 722 cm$^{-1}$; GC-MS (EI): 182 ([M+1]$^+$, 7%), 181 ([M]$^+$, 0.6%), 180 ([M-1]$^+$, 3%), 138 (24%), 124 (36%), 110 (59%), 97 (85%), 82 (56%), 69 (42%), 57 (62%), 41 (100%); HRMS (DART-TOF) $C_{12}H_{23}N$ [M+H]$^+$: calc. 182.1919, found 182.1909.

27. The submitters stored the product at 6 °C in a chemical refrigerator absent of light under an atmosphere of argon for a period of several weeks and observed no apparent decomposition or discoloration. A second sample was stored at room temperature on the laboratory bench in the absence of an argon atmosphere and the product returned to the original light yellow color observed prior to distillation. A check of the refractive index resulted in an $n_D^{20}$ = 1.4355, which is similar to the value obtained prior to distillation.

## Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

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.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

## Discussion

The nitrile functionality is an important motif found within a variety of pharmaceuticals,[10] natural products,[11] agrochemicals,[12] and it is often used to access other functionalities.[13] While there are many ways to prepare nitriles,[14] the vast majority of such methodologies involve a substitution reaction using a cyanide source[15] or a metal-mediated coupling reaction.[14a, 16]

There are few practical procedures for the oxidation of amines to nitriles, and these methods generally involve the use of a transition metal catalyst.[17]

The procedure described above offers a metal-free, scalable, operationally simple method for the oxidation of primary amines to nitriles in good to excellent yield. This overall conversion of an amine to a nitrile involves several intertwined catalytic cycles as described in our original report.[4] By employing catalytic quantities of a commercially available nitroxide catalyst, 4-acetamidoTEMPO, and inexpensive pyridinium bromide as a halide source in conjunction with Oxone, an environmentally benign terminal oxidant, the methodology offers a "green" approach to nitriles.

## Table 1. Substrate Scope of the Oxidation[4]
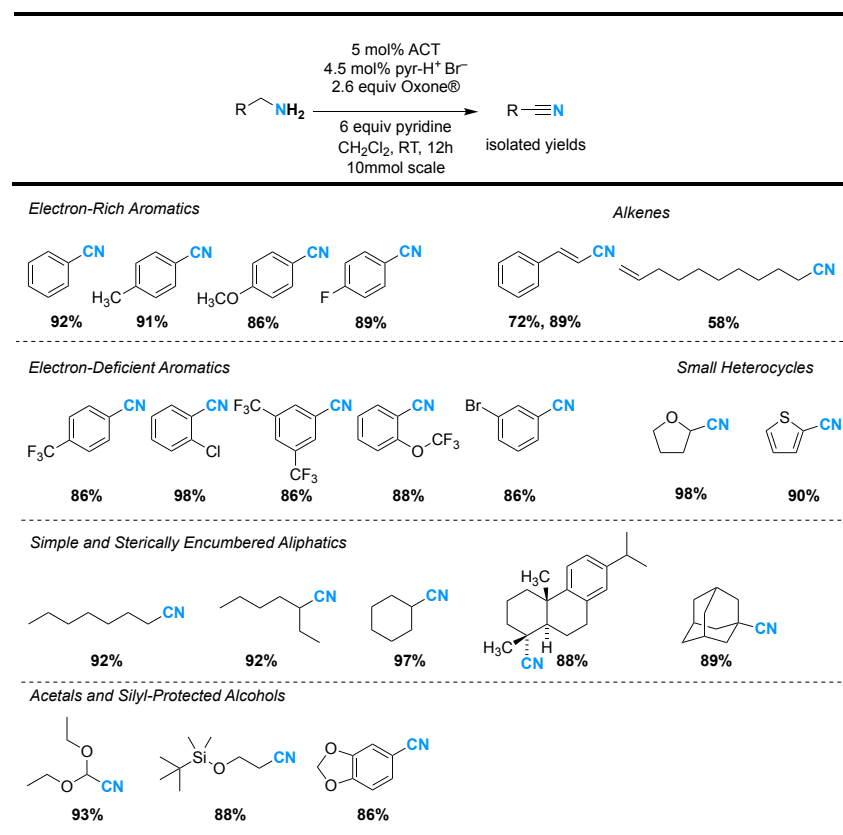
The oxidation is of wide scope as demonstrated by the results presented in Table 1.[4] Additionally, the near neutral pH of the reaction medium coupled with the simple work-up allows for the oxidation of acid-sensitive substrates.

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**Appendix**
**Chemical Abstracts Nomenclature (Registry Number)**

Dodecanenitrile: Dodecanenitrile; (2437-25-4)
Dichloromethane: Methane, dichloro-; (75-09-2)
Oxone: Potassium peroxymonosulfate sulfate; (37222-66-5 or 70693-62-8)
Pyridinium bromide: Pyridine, hydrobromide (1:1); (18820-82-1)
Pyridine: Pyridine; (110-86-1)
4-AcetamidoTEMPO: 1-Piperidinyloxy, 4-(acetylamino)-2,2,6,6-tetramethyl-; (14691-89-5)
4-Acetamido-2,2,6,6-tetramethylpiperidine 1-oxyl: 1-Piperidinyloxy, 4-(acetylamino)-2,2,6,6-tetramethyl-; (14691-89-5)
Dodecylamine: 1-Dodecanamine; (124-22-1)
Pyridine *N*-Oxide; Pyridine, 1-oxide; (694-59-7)

Kyle Lambert received dual B.S. degrees summa cum laude in chemistry and forensic science from the University of New Haven in 2012. He obtained his Ph.D. in 2017 from University of Connecticut while working with Bill Bailey. Kyle's doctoral research involved the exploration of oxoammonium salts as selective oxidants as well as conformational studies of saturated heterocycles. Currently he is completing his postdoctoral studies in John Wood's group at Baylor University in the area of natural product total synthesis, and plans to pursue an academic position upon completion of his postdoctoral studies.

Sherif Eldirany worked as an undergraduate researcher in the Bailey Lab at the University of Connecticut. Sherif's honors thesis focused on exploring novel oxoammonium salt oxidations of amines and in the spring of 2016 he graduated summa cum laude with an honors B.S. degree in chemistry (UCONN). Sherif is currently pursuing a medical degree at the Yale School of Medicine.

Jim Bobbitt received his B.S. degree in chemistry from the University of West Virginia and then obtained his Ph.D. at Ohio State University where he worked with M. L. Wolfrom. Following postdoctoral studies with Carl Djerassi at Wayne State University, Jim was appointed in 1956 to an instructorship in chemistry at the University of Connecticut in Storrs. He rose through the ranks at the University of Connecticut and served as department head from 1976-1982. Jim formally retired in 1992, but has continued to do bench chemistry himself and with a number of colleagues and their graduate students.

![Organic Syntheses logo]

Bill Bailey received his B.S. degree in chemistry in 1968. He worked with Ernest Eliel at the University of Notre Dame investigating conformations of saturated heterocycles and the stereochemical dependence of C-13 shifts and obtained his Ph.D. in 1973. After a two-year postdoc with Kenneth Wiberg at Yale, Bill joined the faculty at the University of Connecticut where he is currently Professor of Chemistry. A major focus of Bill's research is development of new synthetic methodology using novel main-group organometallic chemistry.

Thomas DeVino is from Yorktown Heights, New York. He is an undergraduate at Northeastern University pursuing his B.S. in Chemistry and aims to graduate in 2020. In 2017 he participated in Northeastern's co-op program and worked at Amgen for six months as a synthetic chemist in process development.

Andreas Rötheli is from Zürich, Switzerland and received his B.S. and M.S. degree in chemistry from the Swiss Federal Institute of Technology in Zürich. At ETH, he performed undergraduate research in the laboratory of Prof. Erick M. Carreira, working on natural products total synthesis. He then received his Ph.D. from Harvard University working in the field of organocatalysis with Eric N. Jacobsen. He is currently a scientist in the process development group at Amgen Cambridge, Massachusetts.

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**Dodecanenitrile** *(crude)* **+ Benzyl benzoate** *($^1$H NMR, 500 MHz, CDCl$_3$)*
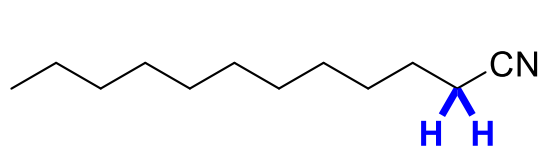


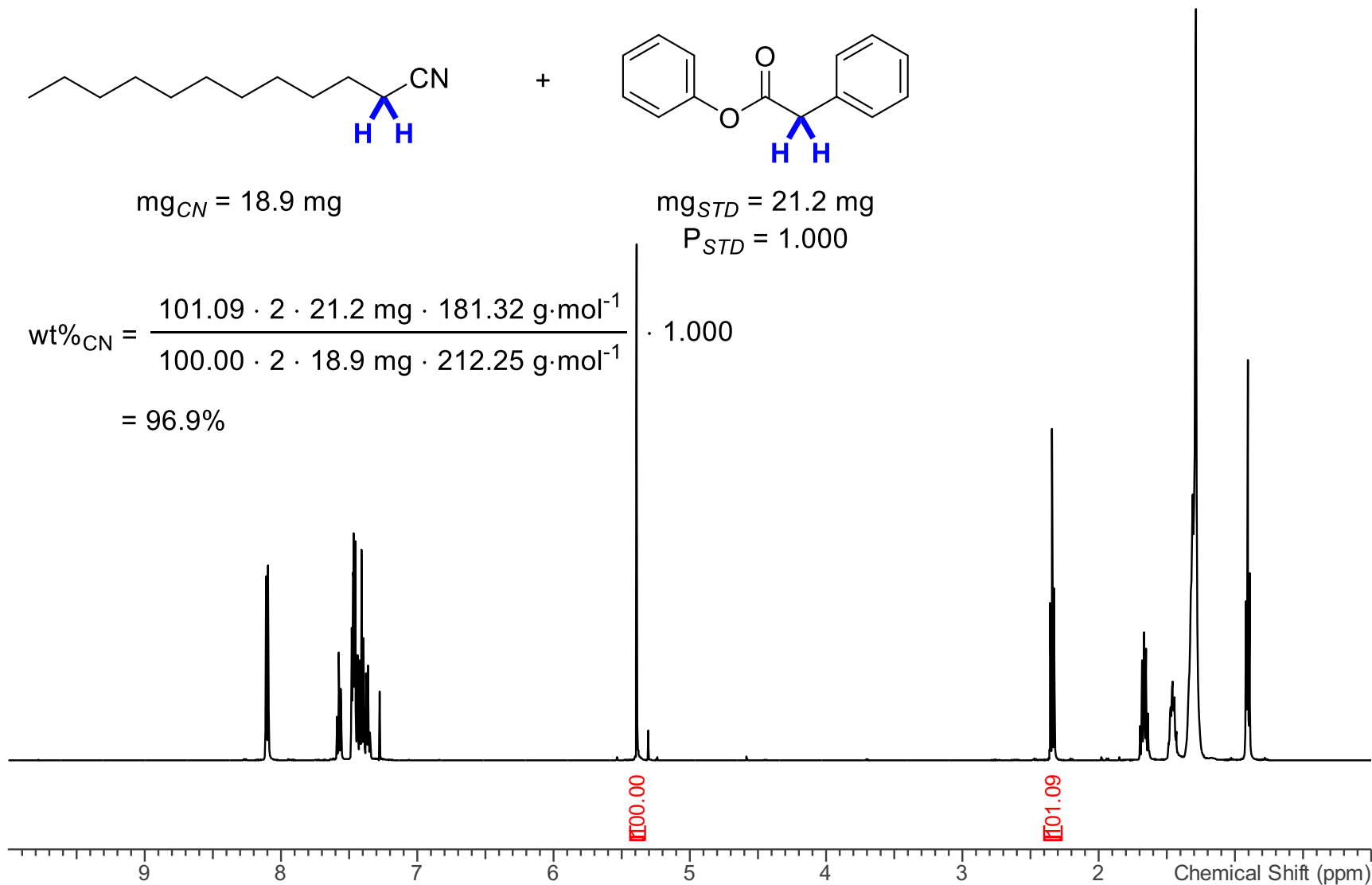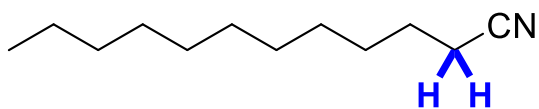$mg_{CN}$ = 18.9 mg
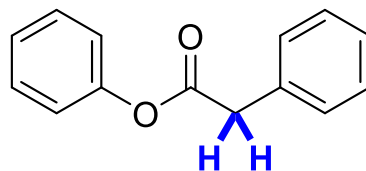
$mg_{STD}$ = 21.2 mg
$P_{STD}$ = 1.000

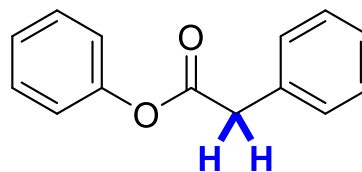$$wt\%_{CN} = \frac{101.09 \cdot 2 \cdot 21.2 \text{ mg} \cdot 181.32 \text{ g·mol}^{-1}}{100.00 \cdot 2 \cdot 18.9 \text{ mg} \cdot 212.25 \text{ g·mol}^{-1}} \cdot 1.000$$

$$= 96.9\%$$

**Dodecanenitrile** *(distilled)* **+ Benzyl benzoate** ($^1$H NMR, 500 MHz, CDCl$_3$)
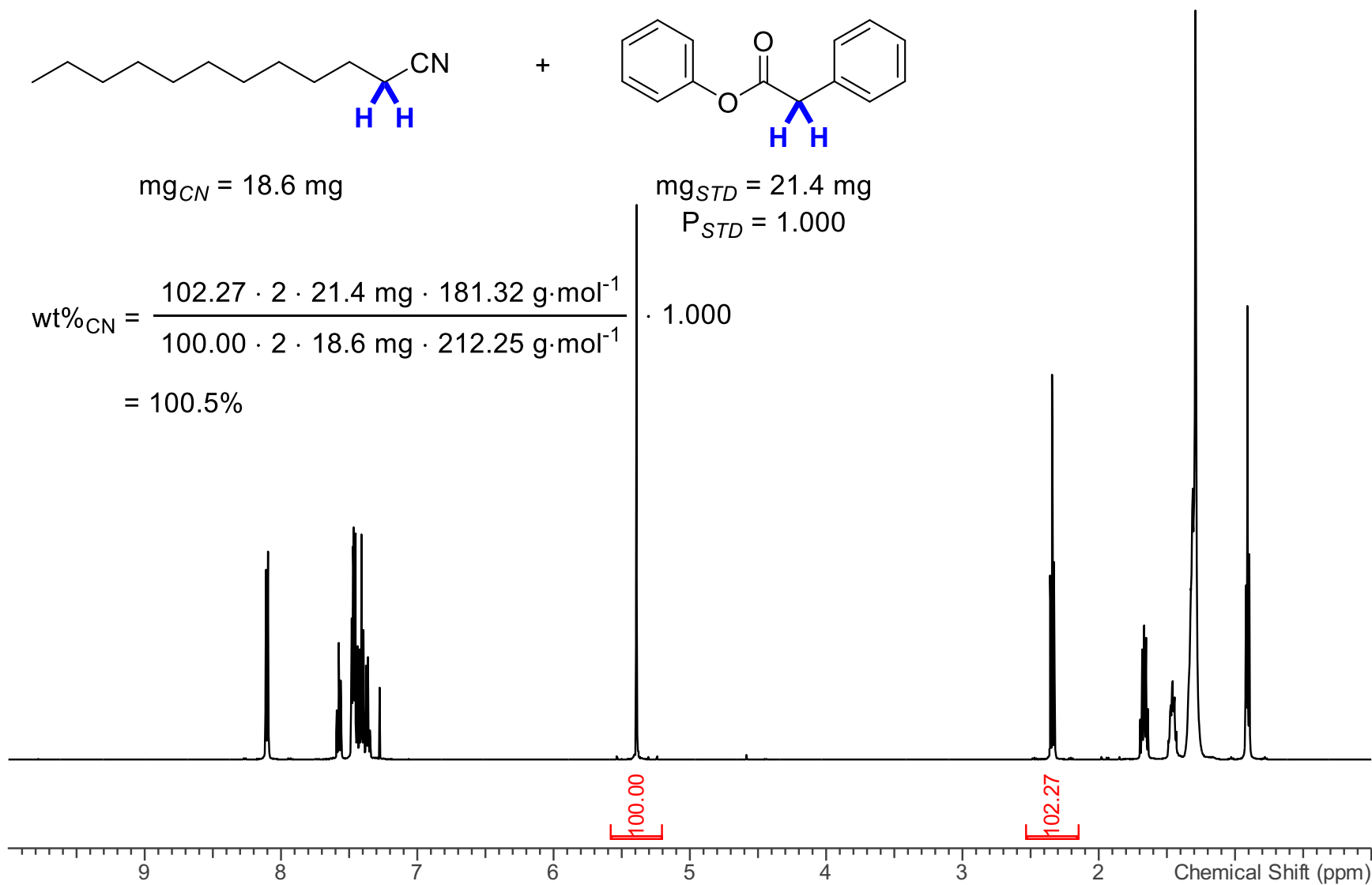


$mg_{CN}$ = 18.6 mg

$mg_{STD}$ = 21.4 mg
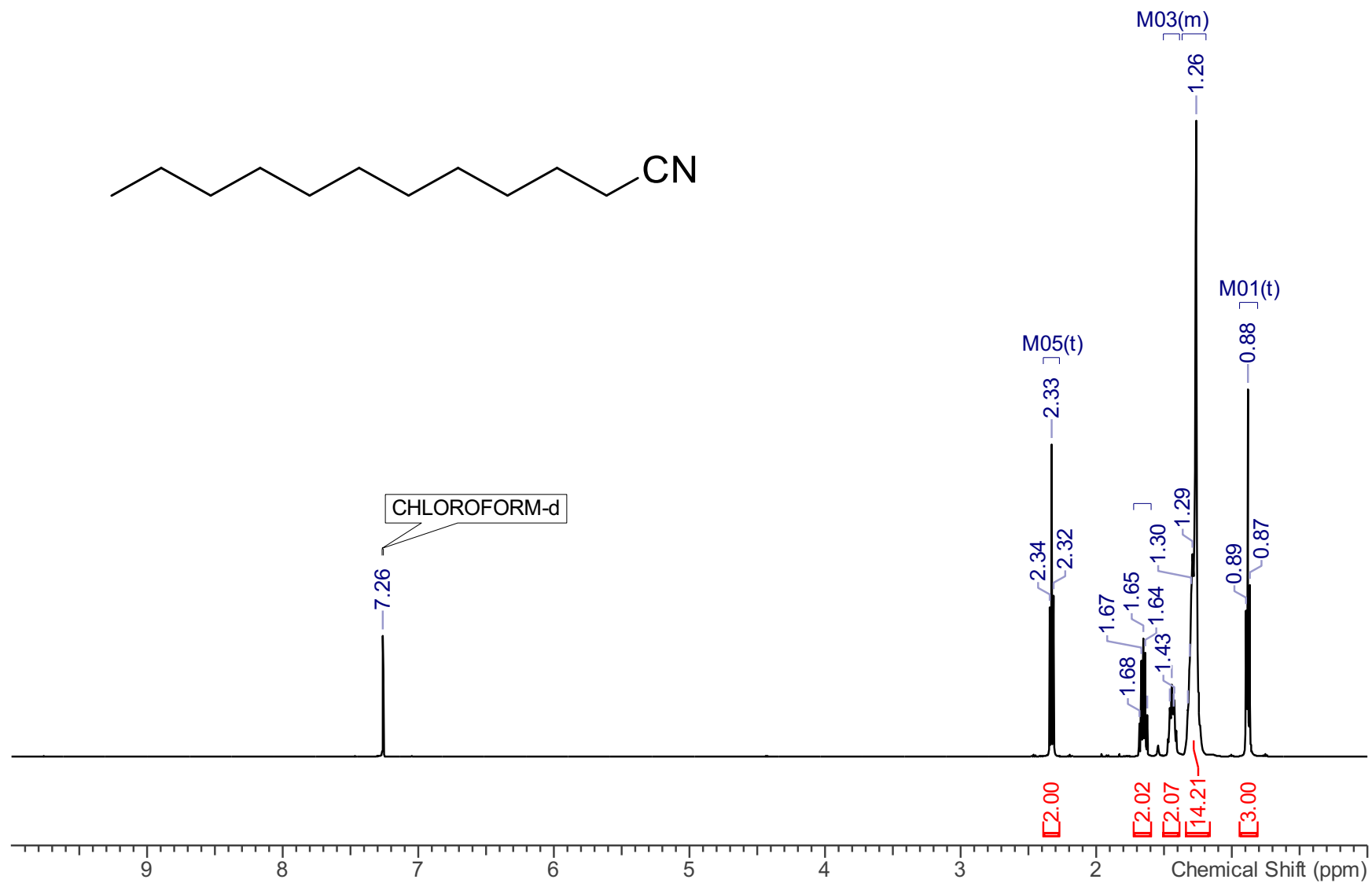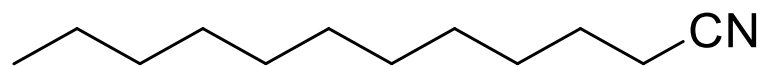$P_{STD}$ = 1.000

$$wt\%_{CN} = \frac{102.27 \cdot 2 \cdot 21.4 \text{ mg} \cdot 181.32 \text{ g·mol}^{-1}}{100.00 \cdot 2 \cdot 18.6 \text{ mg} \cdot 212.25 \text{ g·mol}^{-1}} \cdot 1.000$$

$$= 100.5\%$$

**Dodecanenitrile** *(distilled, $^1$H NMR, 500 MHz, CDCl$_3$)*

**Dodecanenitrile** *(distilled, $^{13}$C NMR, 500 MHz, CDCl$_3$)*