Synthesis of Diethyl (1-Diazo-2-oxopropyl)phosphonate
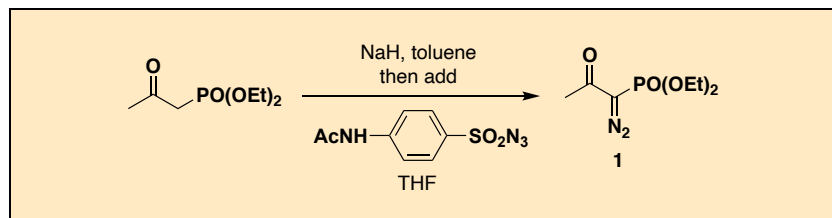
Cholapat Varongchayakul and Rick L. Danheiser*[1]

Department of Chemistry, Massachusetts Institute of Technology, Cambridge, MA 02139

Checked by Michael D. Clift, Steve M. Richter, Zhe Wang, and Seble Wagaw

Procedure (Note 1)

Diethyl (1-diazo-2-oxopropyl)phosphonate (1). A 1-L, three-necked, round-bottomed flask is equipped with a 125-mL pressure-equalizing addition funnel fitted with a rubber septum, an argon inlet adapter, a rubber septum, and a 50 x 20 mm Teflon-coated oval magnetic stir bar (Figure 1A) (Note 2). The flask is charged with NaH (1.04 g, 60% dispersion in mineral oil, 26 mmol, 1.0 equiv) (Notes 3 and 4) and toluene (50 mL) (Note 5). The stirred suspension of NaH is cooled to 0 °C in an ice-water bath, and a solution of diethyl (2-oxopropyl)phosphonate (5.05 g, 26 mmol, 1.0 equiv) (Note 6) in toluene (40 mL) is added dropwise via the addition funnel (Note 7) over 15 min. A white precipitate appears immediately accompanied by bubbling, which subsides after several minutes (Figure 1B). Toluene (10 mL) is charged to the addition funnel and then added dropwise over 2 min to wash any residual phosphonate solution into the reaction flask.
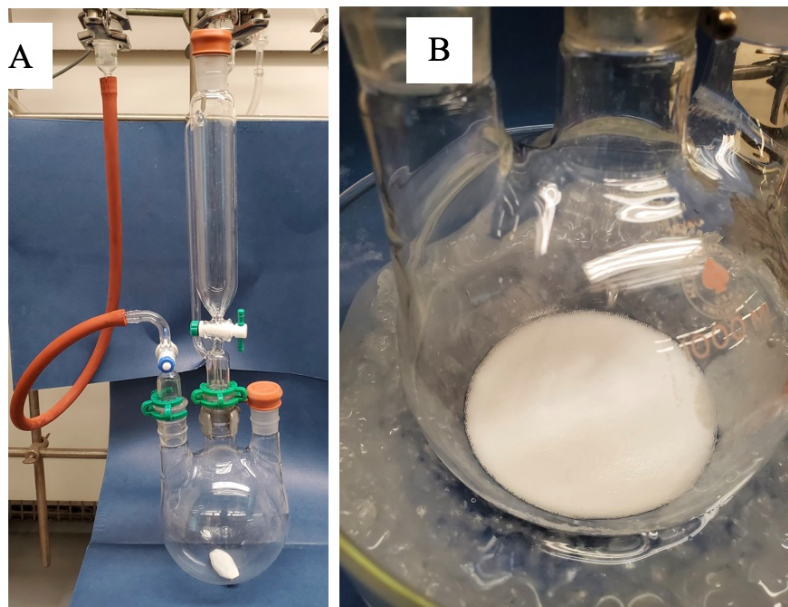
Figure 1. (A) Reaction assembly; (B) Reaction mixture after complete addition of diethyl (2-oxopropyl)phosphonate (photos provided by submitters)

After 10 min, a solution of 4-acetamidobenzenesulfonyl azide (6.25 g, 26 mmol, 1.0 equiv) in 40 mL of THF is added dropwise via the addition funnel over 15 min (Notes 9 and 10). Additional THF (10 mL) is then charged to the addition funnel and added dropwise over 2 min to wash any residual azide solution into the reaction mixture. The ice-water bath is then removed and the reaction mixture is allowed to stir at room temperature for 20 h (Figures 2A-2C). The resulting orange mixture is diluted with hexanes (25 mL) (Note 11) and filtered under vacuum (20 mmHg) through a pad of Celite (20 g) (Note 12) in a 500-mL sintered glass filter funnel into a 1-L Erlenmeyer flask (Figures 3A-3B). The material in the funnel is washed with methyl *t*-butyl ether (3 x 20 mL) (Note 13). The filtrate is next transferred to a 1-L, one-necked, round-bottomed flask (Figure 3C) with the aid of three 10-mL portions of methyl *t*-butyl ether and then concentrated by rotary evaporation (25 °C, 10 mmHg) to yield a pale-yellow oil (7.71 g) (Figure 4) (Note 14).
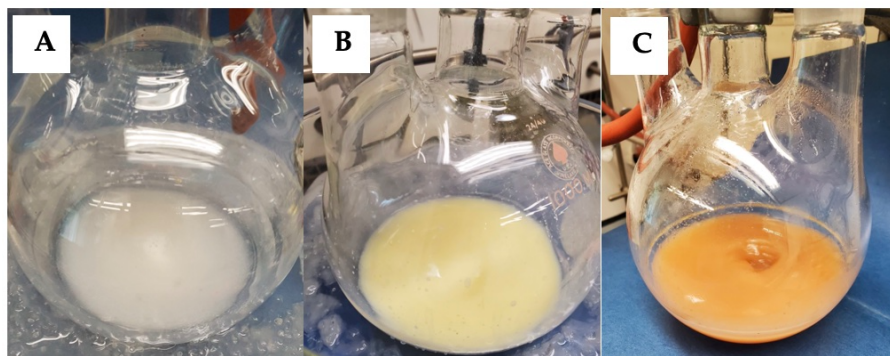
Figure 2. (A) Reaction mixture before addition of 4-acetamidobenzenesulfonyl azide; (B) Reaction mixture after addition of 4-acetamidobenzenesulfonyl azide; (C) Reaction mixture after 20 h
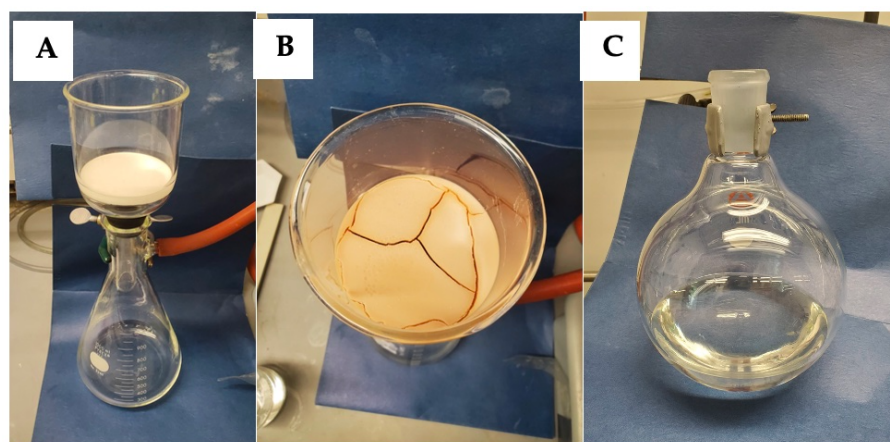(photos provided by submitters)

Figure 3. (A) Filtration set up; (B) Precipitate obtained; (C) Filtrate containing crude reaction mixture in a 1-L round-bottomed flask
(photos provided by submitters)

This material is purified by chromatography on silica (Notes 15 and 16) using 1:1 ethyl acetate-hexanes (Notes 11 and 17). Fractions containing the product are concentrated by rotary evaporation (25 °C, 10 mmHg). Further drying under vacuum (25 °C, 0.05 mmHg) yields 5.17 g (90%) of **1** as a pale-yellow oil (Figure 5B) (Notes 18, 19, 20, 21, and 22).

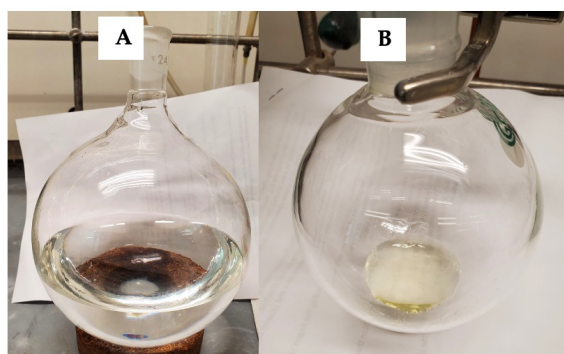**Figure 4. Crude product (photo provided by submitters)**

Figure 5. (A) Fractions containing product; (B) Diethyl (1-diazo-2-oxopropyl)phosphonate (photos provided by submitters)

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

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 sodium hydride, diethyl (2-oxopropyl)phosphonate, 4-acetamidobenzenesulfonyl azide, hexanes, tetrahydrofuran, methyl tert-butyl ether, ethyl acetate, toluene, and silica gel. Differential Scanning Calorimetry (DSC) studies on the reagents and product were performed by the checkers, and the results are summarized in the appendix.

2. Glassware was flame-dried under vacuum (0.1 mmHg), back-filled with argon while hot, and then maintained under the inert atmosphere during the course of the reaction. The checkers did not flame-dry the glassware and obtained similar results reported by the submitters. The relatively large stir bar is required for efficient stirring of the heterogeneous reaction mixture once the 4-acetamidobenzenesulfonyl amide byproduct precipitates from solution. Use of an overhead stirrer is a recommended alternative.

3. Sodium hydride (60% by weight dispersion in mineral oil) was purchased from Oakwood Chemicals and used as received. NaH was weighed into a 20-dram vial and then added to the reaction flask via a powder funnel. The vial was rinsed with 10 mL of toluene. An additional 40 mL of toluene was charged into the reaction flask by syringe resulting in a total of 50 mL of toluene.

4. The use of impure samples of NaH (contaminated with NaOH formed during storage) led to reduced yields and product contaminated with DAMP (diethyl diazomethylphosphonate) presumably generated by cleavage of the product by hydroxide. DAMP is not easily separated from the product by chromatography; the product (1) has $R_f = 0.24$ (silica gel, elution with 1:1 ethyl acetate-hexanes) while DAMP gives a slightly more polar overlapping spot.

5. Anhydrous toluene (Sigma-Aldrich, 99.8%) was used without purification by the checkers.

6. Diethyl 2-(oxopropyl)phosphonate (96%) was purchased from Sigma-Aldrich and used it as received.

7. A solution of diethyl (2-oxopropyl)phosphonate in 30 mL of toluene was prepared in an oven-dried 50-mL, one-necked, round-bottomed flask and transferred to the addition funnel under argon via cannula (Figure 6). The flask was washed with two 5-mL portions of toluene which are cannulated into the addition funnel.

Figure 6. Cannulation set up (photo provided by submitters)

8. 4-Acetamidobenzenesulfonyl azide (97%) was purchased from Oakwood Chemicals and used as received.
9. Anhydrous tetrahydrofuran (>99% with 250 ppm BHT) was purchased from Sigma-Aldrich and used without purification.
10. A solution of 4-acetamidobenzenesulfonyl azide in 30 mL of THF was prepared in an oven-dried 50-mL, one-necked, round-bottomed flask and transferred to the addition funnel under argon via cannula. The flask was washed with two 5-mL portions of THF which are cannulated into the addition funnel.
11. Hexanes (mixture of isomers, >99.7%) was purchased from Sigma-Aldrich and used as received.

12. Filtration through Celite is required to avoid the clogging observed when filtration of the sulfonamide byproduct is attempted using filter paper or a sintered glass funnel. Celite was purchased from EMD Chemicals and used as received.

13. Methyl *tert*-butyl ether (ACS Grade, >99.0%)) was purchased from Sigma-Aldrich and used as received.

14. A second run on identical scale yielded 5.85 g of a pale, yellow oil.

15. Purification was performed using automated column chromatography by the following procedure. The crude product was loaded onto a 40-mm diameter column packed with silica gel (40 g) compatible with CombiFlash Nextgen 100. Elution was carried out with 1:1 ethyl acetate/hexanes (Notes 11 and 17), and 18-mL fractions were collected in 18 x 160 mm test tubes. (Figure 7)
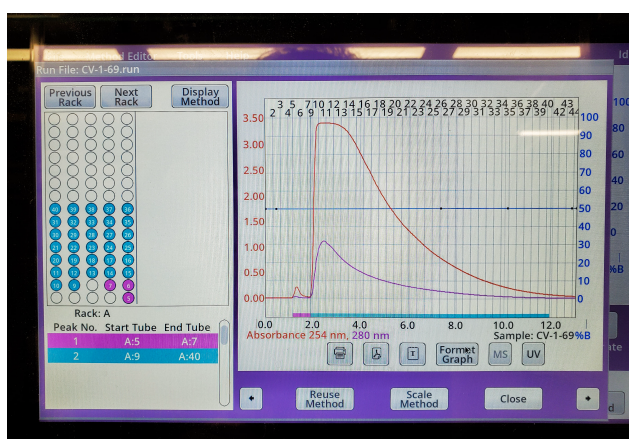
Figure 7. Chromatogram from automated chromatographic separation using CombiFlash Nextgen 100 (photo provided by submitters)

16. Alternatively, the crude product was loaded onto a 60-mm diameter column packed with silica gel (60 g, VWR "Industrial Grade" irregular silica gel 60A, 40-60 μm, 200-300 mesh) that was prepared as a slurry in 1:1 ethyl acetate-hexanes (Notes 11 and 17). Elution was performed with 1:1 ethyl acetate-hexanes, and 20-mL fractions were collected in 18 x 160 mm test tubes with the presence of the desired product monitored by TLC (R_f = 0.24 (1:1 ethyl acetate-hexanes; J.T. Baker silica gel plate). The product was collected in fractions 11-46, which were

combined in a 2-L round-bottomed flask (Figure 5A) and evaporated to dryness using a rotary evaporator (25 °C, 10 mmHg).

17. Ethyl acetate (>99.7%) was purchased from Sigma-Aldrich and used as received.

18. The submitters report that a slightly higher yield of **1** (5.32 g, 93%, 97% wt purity) can be obtained by increasing the amount of diethyl 2-(oxopropyl)phosphonate to 1.1 equiv (5.55 g, 28.6 mmol).

19. Diethyl (1-diazo-2-oxopropyl)phosphonate (**1**) has the following physical and spectroscopic properties: R_f = 0.24 (1:1 ethyl acetate-hexanes; J.T. Baker silica gel plate); ^{1}H NMR (CDCl$_3$, 400 MHz) δ: 4.31–4.10 (m, 4H), 2.29 (s, 3H), 1.40 (td, 6H).; ^{13}C NMR (CDCl$_3$, 100 MHz) δ: 190.1 (d, J = 13.1 Hz), 65.6, 63.4 (d, J = 6.1 Hz), 27.2, 16.1 (d, J = 6.1 Hz).; ^{31}P NMR (CDCl$_3$, 162 MHz) δ: 11.02; FTIR (neat) (cm^{-1}) 2986, 2117, 1655, 1260, 1006. The ^{13}C resonance of the carbon bearing the diazo group, which appears at 65.6 ppm, is likely one peak of a doublet, with the other peak of the doublet buried beneath the resonance at 63.4 ppm. The submitters report the diazo carbon's ^{13}C resonance as 64.1 (d, J = 217 Hz); note, however, that some prior reports do not observe this resonance due to long carbon T1 relaxation times.

20. Diethyl (1-diazo-2-oxopropyl)phosphonate (**1**) is stable to storage neat under air for at least 3 months.

21. The purity of product (**1**) was determined by qNMR analysis on a sample prepared by dissolving 16.8 mg of **1** and 15.1 mg of 1,3,5-trimethoxybenzene (99.9%) in 1.0 mL CDCl$_3$ in a 1 dram vial. The purity was determined to be 97wt%.

22. A second run on identical scale provided 4.97 g (87%) of **1** as a pale-yellow oil.

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

Diethyl and dimethyl (1-diazo-2-oxopropyl)phosphonate **1** and **2** (Scheme 1) are versatile building blocks for organic synthesis.[2] These diazo compounds react via dipolar cycloaddition and other pathways to provide access to diverse classes of nitrogen heterocycles including phosphoryl-substituted pyrazoles, triazolines, oxazoles, and thiazoles.[2] In 1989, Ohira[3] showed that cleavage of the acetyl group can be effected under mildly basic conditions, providing a convenient method for the generation of the Seyferth-Gilbert reagent (diazomethyl)phosphonate ("DAMP")[4] in situ for further transformations. Colvin had previously shown that DAMP reacts with carbonyl compounds in the presence of base to form vinylidene carbenes which are useful synthetic intermediates.[5] Vinylidene carbenes derived from aldehydes rearrange smoothly to alkynes,[5,6] while those generated from ketones can be trapped by alcohols to form enol ethers.[3,7]
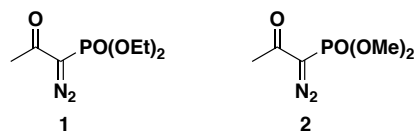
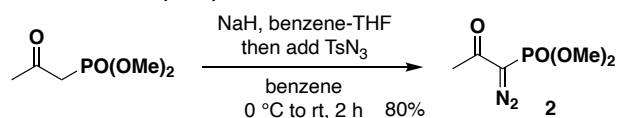**Scheme 1. Diethyl and dimethyl (1-diazo-2-oxopropyl)phosphonate**

The application of **1** and **2** for the one-carbon homologation of aldehydes to alkynes was first reported by Ohira[3] and subsequently developed further by Bestmann.[8] Today the "Ohira-Bestmann reaction" provides one of the most widely used methods for the synthesis of acetylenes.[9] Although (diazomethyl)phosphonate (DAMP) itself is commercially available, DAMP is expensive and has limited shelf life, and the Ohira-Bestmann protocol provides an efficient and convenient means of generating the conjugate base of DAMP in situ for reaction with aldehydes.

Although commercially available, both diethyl and dimethyl (1-diazo-2-oxopropyl)phosphonate are relatively expensive and so many labs opt to prepare the reagents which can then be stored indefinitely for use in the Ohira-Bestmann homologation and other reactions. As summarized in Scheme 2, several groups have reported the application of diazo transfer[10] to dialkyl (2-oxopropyl)phosphonate as an efficient method for the preparation of the "Ohira-Bestmann reagents" **1** and **2**.[11-15]
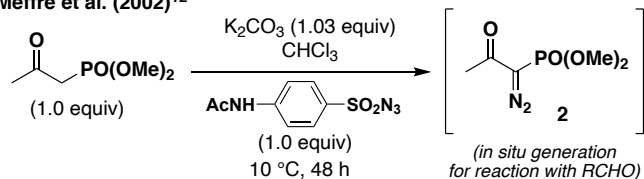
Vandewalle described the first synthesis of **2** via diazo transfer in 1984 using TsN$_3$.[11] The hazards associated with this azide led Meffre[12] and Pietruszka[14] to instead employ 4-acetamidobenzenesulfonyl azide ("p-ABSA"), a sulfonyl azide introduced by Davies[16] in 1987 as a safer alternative to MsN$_3$ and TsN$_3$. Finally, Hanson[13] has deployed a polymeric sulfonyl azide to facilitate separation of the sulfonamide byproduct formed in the reaction, and Kristensen[15] has described the use of an imidazolyl sulfonyl azide for the generation of **2** in situ for reactions with aldehydes in the Ohira-Bestmann alkyne synthesis.

The procedure described in this article employs a modification of the method of Pietruszka.[14] We have found that sodium hydride is a reliable choice as base for the reaction provided that it is not contaminated with NaOH formed by exposure to moisture during storage. Reduced yields of **1** were obtained using NaH from old containers due to cleavage of the acetyl group by nucleophilic hydroxide. Attempts to effect the diazo transfer using DBU in place of NaH led to complex mixtures and reaction with Et$_3$N was very slow and afforded <10% of the desired diazo compound.
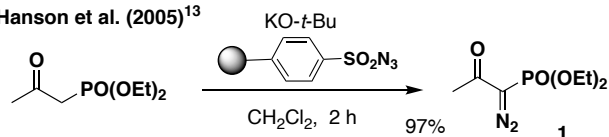
Vandewalle et al. (1984)[11]

NaH, benzene-THF
then add TsN$_3$
benzene
0 °C to rt, 2 h   80%

Meffre et al. (2002)[12]

K$_2$CO$_3$ (1.03 equiv)
CHCl$_3$

AcNH—⟨ ⟩—SO$_2$N$_3$
(1.0 equiv)
10 °C, 48 h

*(in situ generation
for reaction with RCHO)*

Hanson et al. (2005)[13]

KO-*t*-Bu

⬤—⟨ ⟩—SO$_2$N$_3$

CH$_2$Cl$_2$, 2 h   97%

Pietruszka et al. (2006)[14]

NaH (1.0 equiv)
toluene, 0 °C, then add

AcNH—⟨ ⟩—SO$_2$N$_3$
(1.0 equiv)
THF, 16 h   77%

Kristensen et al. (2014)[15]

K$_2$CO$_3$ (3.8 equiv)

⟨N⟩N—SO$_2$N$_3$ • HCl
(1.0 equiv)
CH$_3$CN, 25 °C, 2 h

*(in situ generation
for reaction with RCHO)*

This Work

NaH (1.0 equiv)
toluene, 0 °C, then add

AcNH—⟨ ⟩—SO$_2$N$_3$
(1.0 equiv)
THF, rt, 20 h

86-88% (4.93-5.01 g)
93% (5.55 g) if 1.1 equiv
sulfonyl azide used
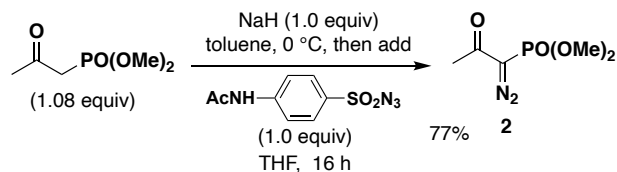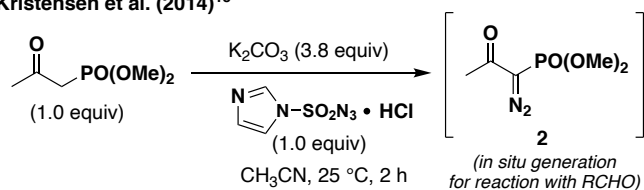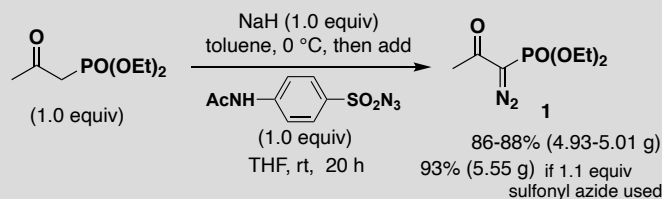
Scheme 2. Synthesis of the Ohira-Bestmann reagents 1 and 2

As the diazo transfer reagent, we favor the use of *p*-ABSA which is commercially available, stable to storage, and safer to handle as compared to other sulfonyl azide reagents. In addition, the sulfonamide byproduct formed in the reaction of this azide can be separated by filtration, simplifying the purification of the diazo product by chromatography.

References

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G.; Bestmann, H. J. Further Improvements of the Synthesis of Alkynes from Aldehydes. *Synthesis* **2004**, 59-62. DOI: 10.1055/s-2003-44346.

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reported to exhibit no impact sensitivity and is a safer reagent for diazo transfer reactions as compared to mesyl azide and tosyl azide.

Appendix
Chemical Abstracts Nomenclature (Registry Number)

Diethyl (2-oxopropyl)phosphonate; (1067-71-6)
Sodium hydride; (7646-69-7)
4-Acetamidobenzenesulfonyl azide; (2158-14-7)

Cholapat Varongchayakul was born in Chonburi, Thailand in 2001. He is currently an undergraduate at the Massachusetts Institute of Technology and expects to receive his B.S. degree in Chemistry in 2024. Cholapat joined the laboratory of Professor Danheiser as an undergraduate researcher in 2020.

Rick L. Danheiser received his undergraduate education at Columbia University where he carried out research in the laboratory of Professor Gilbert Stork. He received his Ph.D. at Harvard University in 1978 working under the direction of E. J. Corey on the total synthesis of gibberellic acid. Dr. Danheiser is the A. C. Cope Professor of Chemistry at MIT where his research focuses on the design and invention of new annulation and cycloaddition reactions, and their application in the total synthesis of biologically active compounds.

Mike Clift is a Principal Research Scientist in the Process Chemistry group at AbbVie. He received his B.S. from Western Michigan University and his Ph. D. from Northwestern University (NU). At NU, he worked with Regan Thomson to develop synthetic methods that were leveraged towards the synthesis of several alkaloid natural products. Mike then moved to Princeton University where he developed a photoredox-organocatalysis platform for C–H functionalization as a post-doctoral scholar with Dave MacMillan. Mike started his independent career at The University of Kansas, where his group developed methods for the synthesis and functionalization of N-containing compounds. Mike joined AbbVie in 2019.

Steve Richter has been in AbbVie Process Engineering for 19 years. He received his BChE from Georgia Tech and his Ph.D. from Purdue University, both in chemical engineering. Since joining industry, Steve has worked process safety and reaction engineering groups in the pharmaceutical and specialty chemical processes.
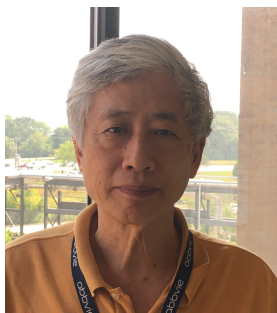
Zhe Wang has been in Abbott/AbbVie Process Safety Laboratory for 25 years. He received his B.S. from Tianjin University in China and his Ph.D. from Northwestern University, both in Chemical Engineering. While at Northwestern, he studied multiphase flow under Professor George Bankoff.

4412330
15062986-760-NMR-1 in CDCL3 $04 BC5129 15mg
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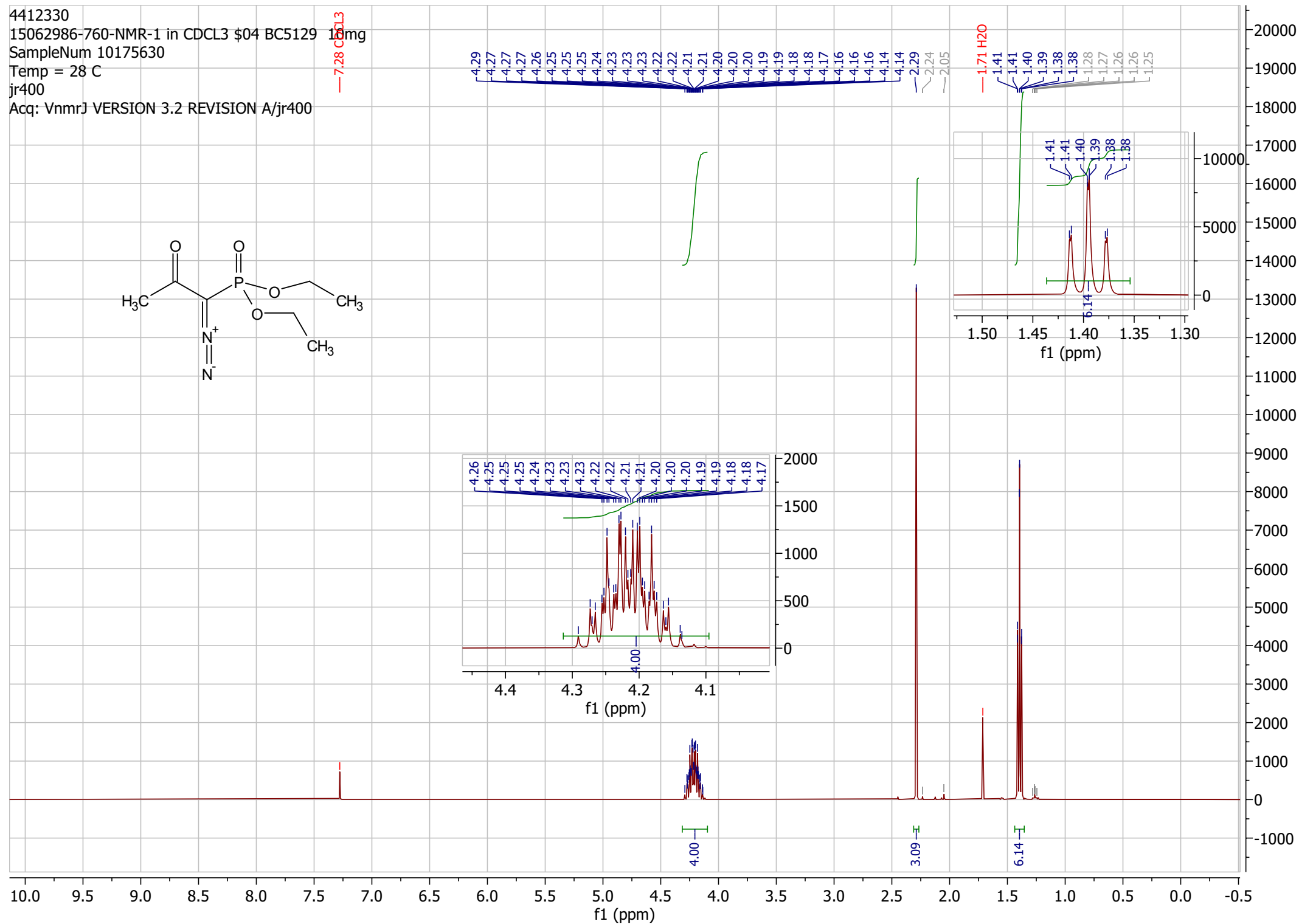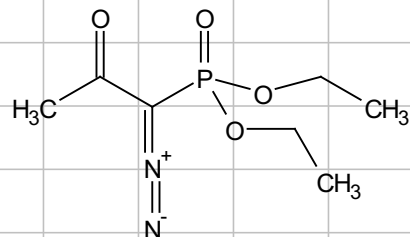
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av400
Acq:TOPSPIN Version 3.5-pl7/av400

11.07
11.02
10.97

f1 (ppm)

Thermal Stability Analysis for Major Reaction Components of Ohira-Bestmann Reagent

Zhe Wang and Steve Richter

The purpose of this study is to screen thermal stability of starting material, reagent and product in the following reaction:

NaH

Summary of Findings and Recommendations

- The starting material diethyl(2-oxopropyl)phosphonate exhibits exothermic events near 246°C (ΔH= -376 J/g) and 278°C (ΔH= -376 J/g).
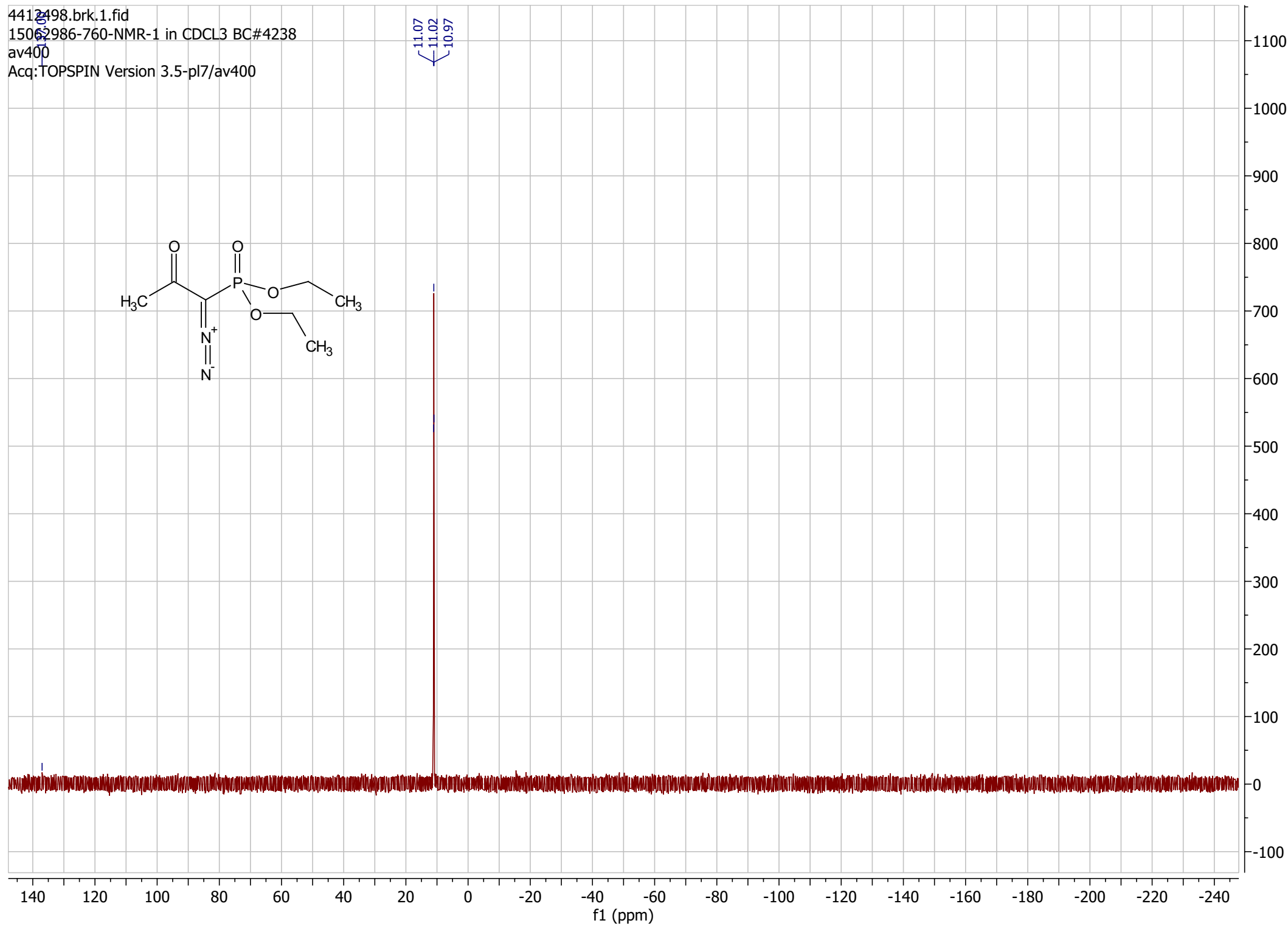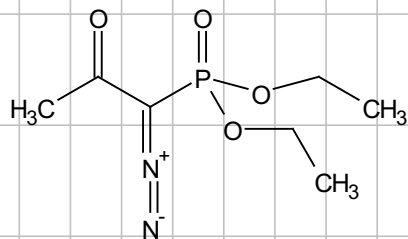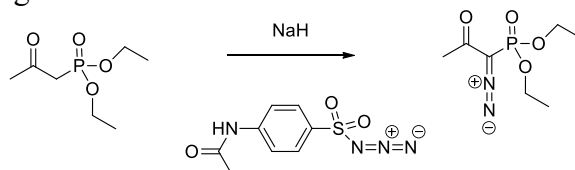- 4-acetamidobenzenesulfonyl azide exhibits an exothermic event near 116°C (ΔH= -1280 J/g) and additional exothermic events near 211°C (ΔH= -17 J/g) and 297°C (ΔH= -145 J/g).
- The product diethyl(1-diazo-2-oxopropyl)phosphonate exhibits exothermic events near 86°C (ΔH= -689 J/g), 226°C (ΔH= -107 J/g) and 276°C (ΔH= -32 J/g).
- In the event that pH is lowered, hydrazoic acid may be formed. The hydrazoic acid is unstable and may decompose violently.

Testing Results

- DSC test results are summarized below:

Compound	ΔH (J/g)	Range (°C)
Diethyl(2-oxopropyl)phosphonate, Oakwood lot 079617N05H, CAS 1067-71-7	-376	246-276
	+37(endo)	276-278
	-659	278-347
4-acetamidobenzenesulfonyl azide, Oakwood lot 450489R06R, CAS 2158-14-7	+91(endo)	95-116
	-1280	116-204
	-17	211-247
	-145	297-336
Product, Diethyl(1-diazo-2-oxopropyl)phosphonate, lot 15062986-0761	-689	86-210
	-107	226-268
	+50(endo)	268-276
	-32	276-295

- The azide reagent, 4-acetamidobenzenesulfonyl azide, exhibits an exothermic event detected near 116°C (ΔH= -1280 J/g).
- The liquid form product, diethyl(1-diazo-2-oxopropyl)phosphonate, exhibits exothermic events near 86°C (ΔH= -689 J/g). The safe handing temperature of the product should be ~100°C below the onset temperature, giving the safety margin for the data obtained using DSC. (Distillation may not be performed without further investigation).
- In the event that pH is lowered, hydrazoic acid may be formed. The hydrazoic acid is unstable and may decompose violently. It has a boiling point of 36°C and a lower explosion limit (LEL) of 10% in nitrogen. The hydrazoic acid may react with metals or organic materials to form unstable metal azides or other organic azides. To prevent the formation of explosive atmosphere in the reactor, nitrogen may need to be flowing through the reactor to keep the hydrazoic acid vapor concentration below 25% of the LEL.

DSC Results

Differential Scanning Calorimetry (DSC) results were acquired in this study with a Mettler Toledo Differential Scanning Calorimeter. Samples were first sealed in gold plated high pressure metal cell or glass cell and heated in the DSC furnace, typically from 0°C to 400°C at 1 – 10°C/min.

Thermal stability of the starting materials, intermediates, and final product was examined by DSC. The results are summarized in Table 1 and the following figures.[1]

Table 1: DSC Results

Compound	Mass (mg)	Test Number	Figure	ΔH (J/g)	Range (°C)
Diethyl(2-oxopropyl)phosphonate, Oakwood lot 079617N05H, CAS 1067-71-7	17.77	23003	1	-376	246-276
				+37(endo)	276-278
				-659	278-347
4-acetamidobenzenesulfonyl azide, Oakwood lot 450489R06R, CAS 2158-14-7	8.85	23001	2	+91(endo)	95-116
				-1280	116-204
				-17	211-247
				-145	297-336
Product, Diethyl(1-diazo-2-oxopropyl)phosphonate, lot 15062986-0761	11.45	23002	3	-689	86-210
				-107	226-268
				+50(endo)	268-276
				-32	276-295

Figure 1: DSC Results for Diethyl(2-oxopropyl)phosphonate

Sample: 23003 (23001) Diethyl(2-oxopropyl)phosphnate, 17.7700 mg

Integral	6685.73 mJ
normalized	376.24 Jg^-1
Left Limit	246.02 ℃
Right Limit	275.64 ℃
Heating Rate	2.00 ℃min^-1

Integral	11.72e+03 mJ
normalized	659.26 Jg^-1
Left Limit	277.74 ℃
Right Limit	347.14 ℃
Heating Rate	2.00 ℃min^-1

Integral	-660.41 mJ
normalized	-37.16 Jg^-1
Left Limit	275.70 ℃
Right Limit	277.74 ℃
Heating Rate	2.00 ℃min^-1

Module: DSC 3+ /700/1928, 05.08.2019 10:29:55
Method: HP M20 Gold Plated, 2℃/min, 0℃-350℃
dt 1.00 s
[1] 0.0..350.0 ℃, 2.00 K/min, N2 50.0 ml/min
Synchronization enabled

Lab: METTLER   STAR* SW 16.10

Figure 2: DSC Results for 4-acetamidobenzenesulfonyl azide

Sample: 23001 (23001) 4-Acetamidobenzenesulfonyl azide, 8.8500 mg

Integral	11.33e+03 mJ
normalized	1279.68 Jg^-1
Left Limit	116.31 ℃
Right Limit	203.98 ℃
Heating Rate	2.00 ℃min^-1

Integral	1286.13 mJ
normalized	145.33 Jg^-1
Left Limit	296.78 ℃
Right Limit	336.46 ℃
Heating Rate	2.00 ℃min^-1

Integral	152.30 mJ
normalized	17.21 Jg^-1
Left Limit	211.23 ℃
Right Limit	247.46 ℃
Heating Rate	2.00 ℃min^-1

Module: DSC3+/700/LN2/1577, 03.01.2019 08:27:11
Method: HP M20 Gold Plated, 2℃/min, 0℃-380℃
dt 1.00 s
[1] 0.0..380.0 ℃, 2.00 K/min, N2 50.0 ml/min
Synchronization enabled

Integral	-805.41 mJ
normalized	-91.01 Jg^-1
Left Limit	95.26 ℃
Right Limit	115.62 ℃
Heating Rate	2.00 ℃min^-1

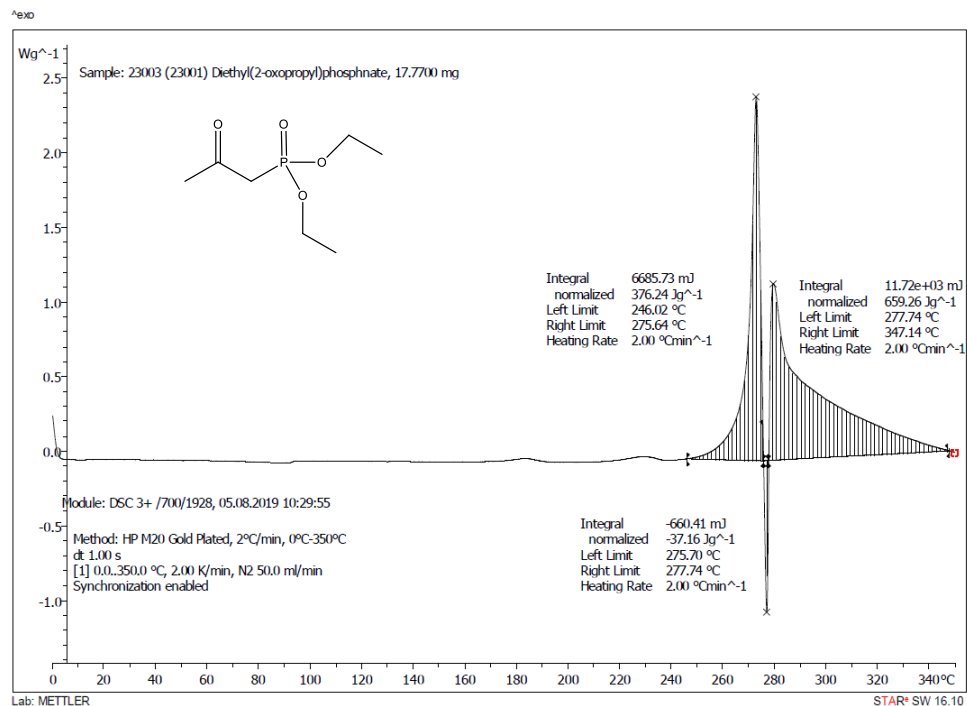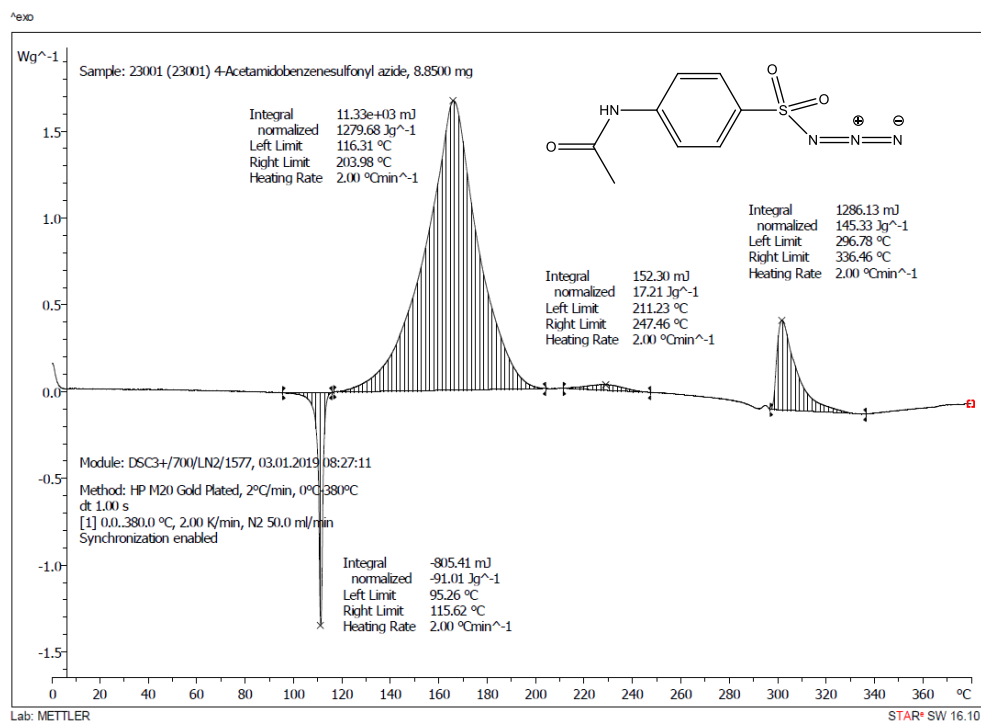Lab: METTLER   STAR* SW 16.10

Figure 3: DSC Results for Diethyl(1-diazo-2-oxopropyl)phosphonate