Selective Monoalkylation of Diethyl Malonate, Ethyl Cyanoacetate, and Malononitrile Using a Masking Group for the Second Acidic Hydrogen

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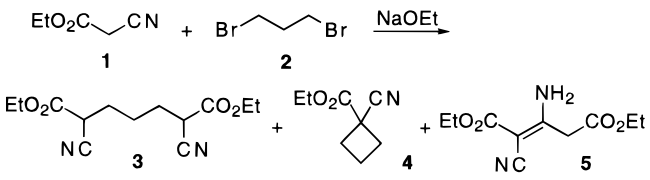
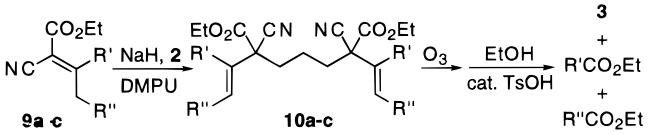
Although the century-old alkylation chemistry of diethyl malonate and related compounds has been somewhat eclipsed over the last 25 years by the use of amide bases, it continues to see wide use in organic synthesis today.[1] For some purposes, e.g., the synthesis of highly functionalized compounds containing quaternary centers[2−4] or the large scale synthesis of small molecules, malonate chemistry remains the method of choice. The selective monoalkylation of diethyl malonate and related compounds can sometimes be problematic, because the monoalkylated products are more nucleophilic than the starting materials. In the case of diethyl malonate, the use of a moderate excess of the nucleophile usually solves the problem nicely, but larger excesses are required for the selective monoalkylation of ethyl cyanoacetate (**1**) because of reduced steric hindrance to formation of the quaternary center, and the problem is even more severe with malononitrile.[1] Dialkylation of **1** can sometimes be avoided by using its tetraalkylammonium salt,[5] or by condensing **1** with the corresponding aldehyde or ketone and reducing the product,[6,7] but these methods are not always applicable.[8] We now report a very simple, general, and environmentally sensitive solution to this problem that is applicable to dialkyl malonates, alkyl cyanoacetates, and malononitrile.

The problem of achieving monoalkylation of **1** became very real for us when we tried to prepare diethyl 2,6-dicyanopimelate (**3**) (Scheme 1). Compound **3** had been reported only once previously, in 1899.[9] It was a byproduct (no yield reported) in the preparation of ethyl 1-cyanocyclobutanecarboxylate (**4**) by alkylation of **1** with 1,3-dibromopropane (**2**). Almost a century later, we were confident that we could find conditions under which **3** could be obtained in good yield from **1** and **2**, especially with the knowledge that tetraethyl 1,1,5,5-pentanetetracarboxylate, the tetraester analog of **3**, could be obtained in about 60% yield from a large excess of diethyl malonate and **2**.[10] To our consternation, however, **3** could not be obtained in more than 12% yield under any conditions that we tried. In general, when a stoichiometric amount of **1** was used, only **4** was obtained; when a large excess of **1** was used, the major product was **5**, derived from self-condensation of **1**.[11−13] These results

Scheme 1



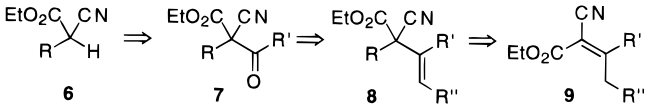
Scheme 2



a: R'= Et, R''= Me. **b:** R'= R''= $(CH_2)_3$. **c:** R'= Me, R''= H.

were consistent through a wide variety of reaction conditions, and other 1,2- and 1,3-dihalides gave similar results. Routes to **3** involving other disconnections proved to be equally unsatisfactory.

It occurred to us that both the cycloalkylation and self-condensation of **1** could be suppressed if one of the acidic hydrogen atoms of **1** was temporarily masked. It was recently reported that the allyl group could serve as a masking group for the second acidic H of diethyl malonate, but its removal required 2 equiv of $(i\text{-PrO})_4$Ti and 4 equiv of *i*-PrMgCl.[14] Other workers recently showed that $EtO_2CCHClCN$ undergoes Michael additions to acrolein, but alkylations of this compound with alkyl halides were not described, and reductive removal of Cl was not demonstrated.[15] We desired a protecting group robust enough to survive alkylation reactions but removable under mild, neutral conditions. We hypothesized that 2-alkylcyanoacetates **6** might be prepared from 2-acyl-2-alkylcyanoacetates **7** by acid- or base-catalyzed alcoholysis and that **7** could in turn be prepared from 2-alkenyl-2-alkylcyanoacetates **8** by ozonolysis. Compounds **8** were first prepared by Cope and Hancock in the 1930s by alkylation of alkylidenecyanoacetates, **9**, exclusively at the α carbon.[16] Ironically, Cope himself ozonolyzed compounds **8** that he synthesized, but he only analyzed the volatile products in order to determine the regioselectivity of the deprotonation of **9**.



We examined the alkylation, ozonolysis, and alcoholysis of several alkylidenecyanoacetates,[17−19] **9a**−**c** (Scheme 2). Alkylations of **9a** and **9b** with **2** proceeded cleanly, giving **10a** and **10b** in 74% and 98% yields, respectively, but **9c** tended to undergo Michael addition to itself. Loss

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(17) Cope, A. C.; Hancock, E. M. *Organic Syntheses*; Wiley: New York, 1955; Collect. Vol. III, p 399.
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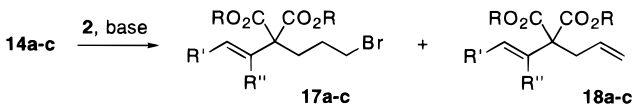
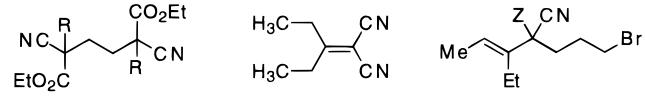
Scheme 3



of CO_2Et from **10a** and **10b** in the course of the alkylation, which Cope sometimes found to be problematic,[16] was completely suppressed by carrying out the reaction in the non-nucleophilic solvent DMPU.

Compounds **10a** and **10b** were then ozonolyzed at −78 °C in CH_2Cl_2. The reaction mixture was allowed to warm to room temperature *without workup*, the solvent was evaporated, and the residue was allowed to reflux overnight in EtOH with catalytic TsOH to give **3** and ester byproducts. The byproduct from the deprotection of **10b**, diethyl glutarate, was difficult to separate from **3**, but the byproducts from the deprotection of **10a**, ethyl acetate and ethyl propionate, were evaporated with the reaction solvent to give **3** in 96% yield *with no purification necessary*.

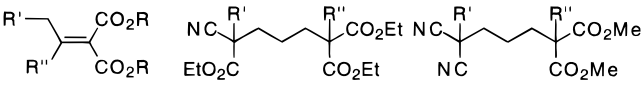
Several compounds analogous to **10** and **3** were also prepared. Alkylation of **9a** with 0.5 equiv of 1,2-dibromoethane (EDB) gave **11a** in 31% yield, and deprotection gave **11b** in 60% yield.[20] The low yield in the alkylation of **9a** with EDB (as compared to **2**) may have been due to side reactions at the much higher temperatures required for it to proceed. An *excess* of **2** was also used to alkylate **9a** and **12**[21] to afford bromides **13a** and **13b** in 62% and 65% yields, respectively. Compounds **13a** and **13b** were then used to alkylate **14a**[22] and **14b**,[23] respectively, to give **15a** and **16a** in 69% and 63% yields. Ozonolysis and alcoholysis then afforded **15b** and **16b** in 78% and 62% yields, respectively.



11a: R= –CEt=CHMe
11b: R= H

12

13a: Z= CO_2Et
13b: Z= CN



14a: R= Et, R'=*i*-Pr, R''= H
14b: R= Me, R'=*i*-Pr, R''= H
14c: R= Et, R'= H, R''= Me

15a: R'= –CEt=CHMe, R''= –CH=CH*i*-Pr
15b: R'= R''= H

16a: R'= –CEt=CHMe, R''= –CH=CH*i*-Pr
16b: R'= R''= H

In contrast to the uneventful synthesis of monobromides **13**, alkylation of alkylidenemalonates[24,25] **14a**−**c** with an excess of **2** always gives large amounts of the allyl derivatives **18a**−**c** in addition to the desired monobromides **17a**−**c** (Scheme 3). It is not certain whether the anion of **14** promotes elimination of HBr from **2** *before* it reacts with **14** or whether elimination occurs from **17** *after* it has already been formed. However, because the alkylations of **14** with **13** proceed smoothly, it seems unlikely that **14** is so much more basic and less nucleophilic than its analogs **9** and **12** that it can induce elimination of HBr from **2**. It therefore must be concluded that **17** is particularly unstable to elimination. This conclusion is supported by the fact that efforts to purify **17** have not been successful. The reason for the instability of **17** is unclear. We point out, though, that the successful syntheses of **15** and **16** suggest that the masking method is normally as applicable to malonate esters as it is to cyanoacetate esters and malononitrile.

In conclusion, we have devised a new masking group for one of the acidic hydrogens of alkyl cyanoacetates, dialkyl malonates, and malononitrile that allows the selective monoalkylation of these nucleophiles. The masking group is distinguished from its alternatives[14,15] by its robustness and the experimental simplicity of its introduction and removal. The environmental innocence of the reagents (3-pentanone and O_3) and the byproducts (H_2O, EtOAc, and EtO_2CEt) and the low cost of the former must also be noted. The method should find application in organic synthesis whenever selective monoalkylation of malonate or its derivatives is desired.

Experimental Section

Standard Procedure for Alkylation. The alkylidenemalonate derivative (2−15 mmol) was added very slowly to a stirring solution of clean NaH (1.1 equiv) in dry DMPU (10−40 mL) under N_2. The bromide (3−45 mmol) was then added, and the reaction was allowed to stir for 2−12 h at a temperature between 20 and 80 °C. The reaction was quenched by adding 1 N HCl (2−15 mL) and diluting with ether. The resulting mixture was extracted four times with water (50−100 mL), dried over $MgSO_4$, and evaporated to give the crude product, which was purified by Kugelrohr distillation and/or flash chromatography.

Diethyl 4,8-Dicyano-3,9-diethyl-2,9-undecadiene-4,8-dicarboxylate (10a). Compound **9a** (5.44 g, 30.0 mmol)[17] was alkylated with a deficiency of 1,3-dibromopropane (2.02 g, 10.0 mmol) at 20 °C overnight using the standard procedure. Unreacted starting material was removed by Kugelrohr distillation. Flash chromatography (10% EtOAc/petroleum ether) gave pure **10a** (2.98 g, 7.41 mmol, 74% yield) as a yellow, oily mixture of diastereomers. ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.86 (q, 6.8 Hz, 1H), 4.25 (q, 7.2 Hz, 2H), 1.85−2.2 (m, 4H), 1.72 (d, 7.0 Hz, 3H), 1.54 (m, 1H), 1.31 (t, 7.2 Hz, 3H), 1.00 (t, 7.6 Hz, 3H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 167.5, 134.6, 125.4, 118.2, 62.8, 55.2, 34.6, 21.2, 20.9, 13.8, 13.6, 13.4. IR (neat): 2965, 2879, 2889, 2246, 1740, 1467, 1228, 1025 cm^{-1}. Anal. Calcd for $C_{23}H_{34}N_2O_4$: C, 68.63; H, 8.51. Found: C, 68.95; H, 8.54.

Diethyl 4,7-Dicyano-3,8-diethyl-2,8-decadiene-4,7-dicarboxylate (11a). Compound **9a** (1.09 g, 6.00 mmol)[17] was alkylated with a deficiency of 1,2-dibromoethane (0.17 mL, 2.00 mmol) at room temperature overnight and then at 70 °C for 2 h using the standard procedure. Unreacted starting material was removed by Kugelrohr distillation. Flash chromatography (10% EtOAc/petroleum ether) gave pure **11a** (0.24 mg, 0.84 mmol, 31% yield) as a yellow, oily mixture of diastereomers. ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.88 (overlapping q's, 1H), 4.26 (m, 2H), 2.16 (m, 4H), 1.73 (overlapping d's, 3H), 1.31 (overlapping t's, 3H), 1.00 (t, 3H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 167.3/167.2, 134.5/134.3, 126.0/125.8, 118.0, 63.1, 54.8, 31.0/30.7, 21.1/20.9, 13.9/13.8, 13.5/13.4. IR (KBr): 2978, 2940, 2243 (w), 1744, 1468, 1229, 1026 cm^{-1}. Anal. Calcd for $C_{22}H_{32}N_2O_4$: C, 68.01; H, 8.30. Found: C, 68.29; H, 8.50.

Ethyl 2-Cyano-2-(3-bromopropyl)-3-pentenoate (13a). Compound **9a** (1.81 g, 10.0 mmol)[17] was alkylated with an excess of 1,3-dibromopropane (6.06 g, 30.0 mmol) at 20 °C overnight using the standard procedure. Unreacted starting material was removed by Kugelrohr distillation. Flash chromatography (10% EtOAc/petroleum ether) gave pure bromide **13a** (1.87 g, 61.9 mmol, 62% yield) as a pale yellow oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.88 (q, 6.8Hz, 1H), 4.26 (q, 7.2Hz, 2H), 3.44 (~q, 2H), 2.24−1.93 (m, 6H), 1.73 (d, 6.8 Hz, 3H), 1.31 (t, 7.0 Hz, 3H), 1.01 (t, 7.6 Hz, 3H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 167.3, 134.5, 125.4, 118.1, 62.8, 54.7, 33.7, 32.3, 28.3, 20.8, 13.7, 13.6,

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13.3. IR (neat): 2975, 2878, 2242, 1740, 1458, 1231 cm^{-1}. Anal. Calcd for $C_{13}H_{20}BrNO_2$: C, 51.67; H, 6.67. Found: C, 52.00; H, 6.81.

7-Bromo-3-ethyl-2-heptene-4,4-dicarbonitrile (13b). Compound **12** (2.14 mL, 15.0 mmol)[21] was alkylated with an excess of 1,3-dibromopropane (4.60 mL, 45.0 mmol). The reaction was allowed to stir at 20 °C for 2 h using the standard procedure. The product was purified by Kugelrohr distillation to give bromide **13b** (2.49 g, 9.76 mmol, 65% yield), 97% pure by GC/MS, as a pale yellow oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 6.03 (q, 6.8 Hz, 1H), 3.47 (t, 5.8 Hz, 2H), 2.06−2.32 (m, 6H), 1.78 (d, 7.0 Hz, 3H), 1.17 (t, 7.4 Hz, 3H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 131.6, 127.7, 114.6, 43.2, 36.9, 31.2, 28.2, 21.2, 13.8, 13.6. IR (neat): 2974, 2247, 1456, 1262 cm^{-1}.

Triethyl 4-Cyano-3-ethyl-11-methyl-2,9-dodecadiene-4,8,8-tricarboxylate (15a). Compound **14a** (0.51 g, 2.00 mmol)[22] was alkylated with **13a** (0.55 g, 2.40 mmol) at 80 °C for 6 h under standard conditions. Flash chromatography (5−15% EtOAc/petroleum ether gradient) gave **15a** (0.45 g, 1.00 mmol, 50% yield) as a colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.90 (dd, 16.2 Hz, 1.4Hz, 1H), 5.81 (q, 6.8 Hz, 1H), 5.48 (dd, 16.2 Hz, 6.8 Hz, 1H), 4.21 (m, 6H), 2.32 (d of octet, J_{octet} = 6.8 Hz, J_d = 1.0 Hz, 1H), 2.14−2.00 (m, 6H), 1.68 (d, 6.8 Hz, 3H), 1.24 (m, 11H), 0.96 (m, 9H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 170.6, 167.7, 139.8, 134.8, 125.2, 123.4, 118.3, 62.7, 61.3, 58.7, 55.4, 35.2, 35.0, 31.3, 22.1, 20.9, 20.2, 14.0, 13.9, 13.7, 13.5. IR (neat): 2965, 2242 (weak), 1732, 1230 cm^{-1}. Anal. Calcd for $C_{25}H_{39}NO_2$: C, 66.79; H, 8.74. Found: C, 66.46; H, 8.88.

Dimethyl 4,4-Dicyano-3-ethyl-11-methyl-2,9-dodecadiene-8,8-dicarboxylate (16a). Compound **14b** (1.20 g, 6.00 mmol)[23] was alkylated with **13b** (1.28 g, 5.00 mmol) at 20 °C overnight under standard conditions. Flash chromatography (2.5−10% EtOAc/petroleum ether gradient) gave **16a** (0.98 g, 3.11 mmol, 62% yield) as a colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 6.00 (q, 6.8 Hz, 1H), 5.90 (dd, 16.1 Hz, 1.4 Hz, 1H), 5.51 (dd, 16.4 Hz, 6.8 Hz, 1H), 3.74 (s, 6H), 2.12−2.25 (m, 3H), 1.97−2.08 (m, 4H), 1.76 (d, 6.8 Hz, 3H), 1.42−1.59 (m, 2H), 1.14 (t, 7.4 Hz, 3H), 1.00 (d, 6.8 Hz, 6H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 170.9, 140.3, 131.8, 127.4, 123.0, 114.7, 58.8, 52.7, 43.8, 38.2, 34.5, 31.3, 22.1, 21.1, 20.5, 13.7, 13.7. IR (neat): 2957, 2874, 2246 (weak), 1736, 1252 cm^{-1}. Anal. Calcd for $C_{21}H_{30}N_2O_4$: C, 67.36; H, 8.07. Found: C, 67.43; H, 8.22.

Standard Procedure for Deprotection. The protected malonate derivative (2−10 mmol) was dissolved in methylene chloride (ca. 30 mL), and the solution was cooled to −78 °C. The solution was sparged with ozone (80 V, 1 L/min) until it turned blue. It was allowed to warm to room temperature, and the solvent was evaporated. The residue was redissolved in EtOH, and 15 mol % $TsOH·H_2O$ was added. The mixture was allowed to reflux overnight. The solvent was evaporated, and ether was added. The solution was shaken with dilute aqueous $NaHCO_3$ and water, dried over $MgSO_4$, and evaporated to give the crude product.

Diethyl 1,5-Dicyanopentane-1,5-dicarboxylate (Diethyl 2,6-dicyanopimelate) (3). Compound **10a** (2.9 g, 7.2 mmol) was deprotected using the standard procedure to give **3** (1.8 g, 6.9 mmol, 96% yield)[9] as a yellow, oily mixture of diastereomers, 97% pure by GC/MS. ^{1}H NMR (200 MHz, $CDCl_3$): δ 4.29 (q, 7.2 Hz, 2H), 3.54 (t, 6.8 Hz, 1H), 2.03 (q, 7.4 Hz, 2H), 1.72 (m, 1H), 1.34 (t, 7.2 Hz, 3H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 165.6, 116.0, 63.0, 37.0, 28.7, 23.9, 13.8. IR (neat): 2984, 2941, 2250 (w), 1744, 1464, 1370, 1256, 1203,1097, 1024, 856 cm^{-1}.

Diethyl 1,4-Dicyanobutane-1,4-dicarboxylate (diethyl 2,6-dicyanoadipate) (11b). Compound **11a** (243 mg, 0.62 mmol) was deprotected using the standard procedure. Flash chromatography (10−30% EtOAc/petroleum ether gradient) gave **11b** (94 mg, 0.37 mmol, 60% yield)[20] as a white solid, mp 69−71 °C, and as an inseparable mixture of diastereomers. ^{1}H NMR (200 MHz, $CDCl_3$): δ 4.31 (q, 7.2 Hz, 2H), 3.61 (m, 1H), 2.17 (m, 2H), 1.35 (t, 7.2 Hz, 3H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 165.0, 115.5, 63.3, 36.7, 26.6, 14.0. IR (KBr): 2987, 2904, 2251 (w), 1725, 1278, 1019, 855 cm^{-1}.

Triethyl 5-Cyanopentane-1,1,5-tricarboxylate (15b). Compound **15a** (0.50 g, 1.11 mmol) was ozonolyzed by the standard method. Flash chromatography (30% EtOAc/petroleum ether) gave **15b** (0.19 g, 0.68 mmol, 61% yield) as a yellow oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 4.32−4.16 (m, 6H), 3.52 (t, 7.2 Hz, 1H), 3.34 (t, 7.2 Hz, 1H), 1.97 (m, 4H), 1.58 (m, 2H), 1.30 (m, 9H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 168.9, 165.8, 116.2, 62.8, 61.5, 51.5, 37.2, 29.3, 27.7, 24.5, 14.0, 13.9. IR (neat): 2983, 2940, 2873, 2250, 1736, 1464, 1370, 1248, 1155, 1097, 1027 cm^{-1}. Anal. Calcd for $C_{15}H_{23}NO_6$: C, 57.20; H, 7.40. Found: C, 57.52; H, 7.58.

Dimethyl 5,5-Dicyanopentane-1,1-dicarboxylate (16b). Compound **16a** (500 mg, 1.33 mmol) was ozonolyzed by the standard method. Flash chromatography (10−30% EtOAc/petroleum ether gradient) gave **16b** (197 mg, 0.83 mmol, 62% yield) as a colorless oil. ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.81 (t, partly obscured, ~11.0 Hz, 1H), 3.76 (s, 6H), 3.40 (t, 7.2 Hz, 1H), 2.03 (m, 4H), 1.66 (m, 2H). ^{13}C{H} NMR (50 MHz, $CDCl_3$): δ 169.1, 112.3, 52.7, 50.9, 30.4, 27.3, 24.2, 22.4. IR (neat): 2956, 2256, 1732, 1437, 1244, 1160 cm^{-1}. Anal. Calcd for $C_{11}H_{14}N_2O_4$: C, 55.46; H, 5.92. Found: C, 55.22; H, 6.08.

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