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Sodium Tetramethoxyborate: An Efficient Catalyst for Michael Additions of Stabilized Carbon Nucleophiles

Araceli G. Campaña,[†] Noelia Fuentes,[†]
Enrique Gómez-Bengoa,[‡] Cristina Mateo,[‡] J. Enrique Oltra,[†]
Antonio M. Echavarren,*[,‡,§] and Juan M. Cuerva*[,†]

Department of Organic Chemistry, Faculty of Sciences, Campus Fuentenueva s/n, University of Granada, E-18071 Granada, Spain, Department of Organic Chemistry, Universidad Autónoma de Madrid, Cantoblanco, 28049 Madrid, Spain, and Institute of Chemical Research of Catalonia (ICIQ), Av. Països Catalans 16, 43007 Tarragona, Spain

aechavarren@iciq.es; jmcuerva@ugr.es

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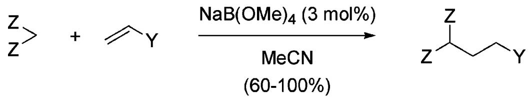


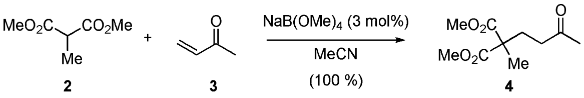
Z, Y = electron-withdrawing groups

Sodium tetramethoxyborate, easily prepared by reaction of inexpensive sodium borohydride with methanol, possesses a suitable combination of a Lewis base and a Lewis acid to catalyze Michael reactions at room temperature under practically neutral conditions. This reaction provides good to excellent yields of Michael addition products from a broad scope of Michael donor and Michael acceptor reagents.

Michael reaction is one of the cornerstones of organic synthesis and is widely used in C−C bond-forming reactions.[1] Nevertheless, conventional base-catalyzed processes are usually affected by undesirable side reactions such as retrograde Michael reactions or polymerizations of the Michael acceptor.[1] Many catalysts working under neutral conditions have been developed to solve these problems.[2] Transition metals play an important role in this regard, especially Ru-based catalysts.[3,4] It should be noted that some phosphines are excellent neutral catalysts for the Michael addition of stabilized carbon nucleopiles,[3,5,6] although the simplest and most stable ones (triphenylphospine, for example) are not reactive enough. The superior chemical profile of some ruthenium catalysts is due to a combination of the presence of phospines and the metal center, the latter acting as a Lewis acid.[3] Encouraged by this idea, we thought that other combinations of a base and a Lewis acid might result in simple, inexpensive, practical, air-stable catalysts that might work under practically neutral conditions. Boron derivatives fit these requirements, their catalytic activity toward the Michael addition of stabilized carbon nucleophiles having been observed previously by us[3] and other authors.[7] We describe here our results in a new and efficient Michael addition reaction catalyzed by simple $NaB(OMe)_4$ (**1**).

Boron derivative **1**, a stable white solid, was easily prepared by reaction of $NaBH_4$ with methanol.[8] Its structure was confirmed by the singlet signal at 3.02 ppm (CD_3OD) observed in its ^{11}B NMR spectrum, which is similar to that described for the closely related reagent $LiB(OMe)_4$ (2.7 ppm, CD_3OD).[9,10]

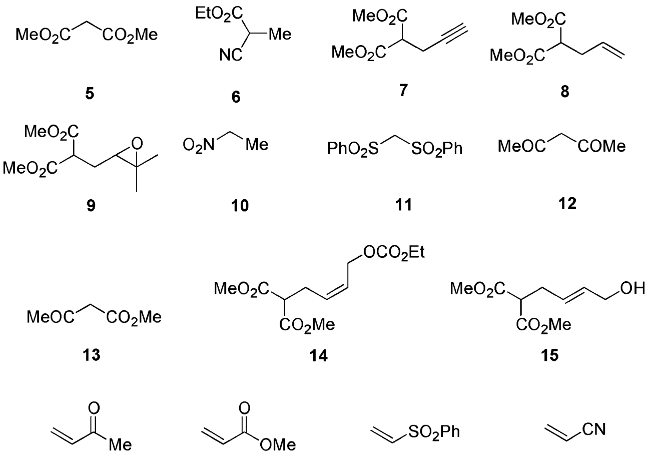
We began to evaluate the catalytic activity of **1** by stirring a substoichiometric proportion of this catalyst (3 mol %) with a mixture of malonate **2** (1 mmol) and methyl vinyl ketone (**3**) (1.1 mmol) in acetonitrile at room temperature (eq 1).[11] In this manner, we obtained a 100% yield of the Michael addition product **4**.



(1)

This excellent result prompted us to study the scope of the reaction using different Michael donors (**5**−**15**) and Michael acceptors (**3**, **16**−**18**) (Chart 1). The results obtained are summarized in Table 1.

CHART 1



As we expected, with the use of different combinations of Michael donors and acceptors, the reaction gave good to excellent yields of the corresponding Michael addition products. Moreover, the presence of common functional groups in the Michael donor such as alkenes, terminal alkynes, nitriles,

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[†] University of Granada.

[‡] Universidad Autónoma de Madrid.

[§] Institute of Chemical Research of Catalonia.

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(2) For selected examples, see: (a) Antonioletti, R.; Bonadies, F.; Monteagudo, E. S.; Scettri, A. *Tetrahedron Lett.* **1991**, *32*, 5373−5374. (b) Ranu, B. C.; Bhar, S.; Sarkar, D. C. *Tetrahedron Lett.* **1991**, *32*, 2811−2812. (c) Bonadies, F.; Lattanzi, A.; Orelli, L. R.; Pesci, S.; Scettri, A. *Tetrahedron Lett.* **1993**, *34*, 7649−7650. (d) Nelson, J. H.; Howells, P. N.; DeLullo, G. C.; Landen, G. L. *J. Org. Chem.* **1980**, *45*, 1246−1249. (e) Keller, E.; Feringa, B. L. *Tetrahedron Lett.* **1996**, *37*, 1879−1882. (f) Veldurthy, B.; Clacens, J. M.; Figueras, F. *Adv. Synth. Catal.* **2005**, *347*, 767−771.

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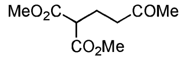
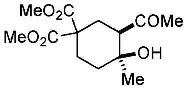
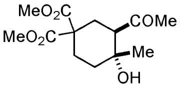
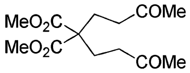
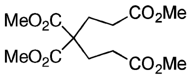
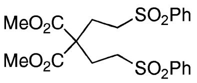
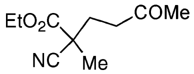
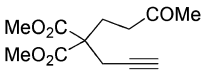
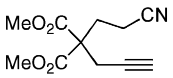
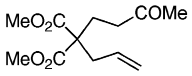
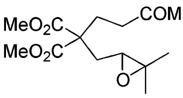
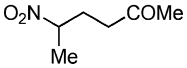
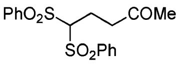
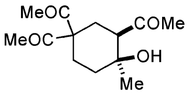
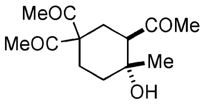
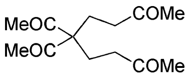
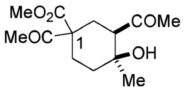
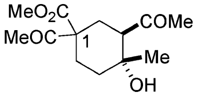
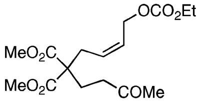
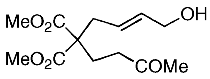
TABLE 1. NaB(OMe)$_4$-Catalyzed Michael Addition of Activated Carbon Nucleophiles[a]

Entry	Michael donor	Michael acceptor	Reaction time (h)	Product	Yield (%)
1	**5**	**3**[b]	3	 **19**	71
2	**5**	**3**	3	 **20a** +  **20b** +  **21**	80 20[c]
3	**5**	**16**	12	 **22**	91
4	**5**	**17**	12	 **23**	82
5	**6**	**3**	3	 **24**	90
6	**7**	**3**	3	 **25**	100
7	**7**	**18**	3	 **26**	69
8	**8**	**3**	3	 **27**	94
9	**9**	**3**	3	 **28**	82
10	**10**	**3**[b]	3	 **29**	78
11	**11**	**3**[b]	24	 **30**	60
12	**12**	**3**	3	 **31a** +  **31b** +  **32**	59 31[d]
13	**13**	**3**	3	 **33a** +  **33b**	61[e] 39[f]
14	**14**	**3**	3	 **34**	91
15	**15**	**3**	3	 **35**	90

[a] Unless otherwise stated the reactions were carried out in the presence of 3 mol % of NaB(OMe)$_4$ using 2 equiv of Michael acceptor and 1 equiv of Michael donor in MeCN at 23 ×bcC. [b] The reaction was carried out using 1 equiv of Michael acceptor. [c] **20b** and **21** were obtained as a 2:1 mixture. [d] **31b** and **32** were obtained as a 4:1 mixture. [e] **33a** was obtained as a 3:2 mixture of epimers at C-1. [f] **33b** was obtained as a 1:1 mixture of epimers at C-1.

epoxides, or alcohols did not interfere with the catalyst (entries 5−9, 14, and 15). Entries 1 and 2 show that either simple or double additions can be alternatively achieved by choosing the suitable proportion of the Michael acceptor reagent. Double addition products, however, can undergo subsequent sodium tetramethoxyborate-catalyzed intramolecular aldol reactions yielding cyclic products such as **20**, **31**, and **33** (entries 2, 12, and 13) with modest stereoselection. Michael donor **15**, which

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has two nucleophilic positions, only gave Michael adduct **35**. This chemoselectivity can be related to differences in acidity between the 1,3-diester ($pK_a = 16$, DMSO)[12] and alcohol ($pK_a = 29$, DMSO)[12] functionalities, the Michael reaction being effective from the most acidic one. Nevertheless, a direct correlation between the acidity of the Michael donor reagent and the reaction rate is not always to be found. In fact bis(phenylsulphonyl)methane **11** ($pK_a = 12.25$, DMSO),[12] which is more acidic than dimethyl malonate **5** ($pK_a = 16$, DMSO),[12] required a much longer reaction time. Control experiments showed that the reaction between malonate **5** (1.0 mmol) and methyl vinyl ketone **3** (2.0 mmol) was complete in 30 min (**20**−**21**, 100%), whereas a 30-min reaction between disulfone **11** (1.0 mmol) and the same Michael acceptor (**3**, 2.0 mmol) only gave traces of the Michael adduct **30**. This intriguing behavior suggests that $NaB(OMe)_4$-catalyzed Michael reactions are not simple base-catalyzed reactions. It is possible that a boron derivative, probably $B(OMe)_3$,[13,14] is acting as a Lewis acid activating not only the Michael acceptor but also the Michael donor toward the base,[15] thus facilitating an efficient Michael addition reaction even with less acidic substrates as diesters.

In summary, $NaB(OMe)_4$ (**1**), easily prepared by reaction of inexpensive sodium borohydride with methanol, possesses an ideal combination of a Lewis base and a Lewis acid to catalyze Michael additions of stabilized carbon nucleophiles. The reaction takes place at room temperature under practically neutral conditions and affords good to excellent yields of Michael addition products with a broad scope of Michael donors and acceptors. This procedure gives access to a variety of functionalized substrates of the type commonly used in studies about new cyclization reactions. Moreover, the inclusion of chiral ligands in the boron-based catalyst might lead to enantioselective Michael reactions.[16] We are currently working toward this goal.

Experimental Section

General. Dry MeCN was obtained by distillation under Ar from CaH_2. Substances **9**[17] and **15**[18] were prepared according to known procedures. The following known compounds were isolated as pure samples and showed NMR spectra identical to those in our previously reported data: **4**, **19, 20a-b, 21, 22, 23, 24, 25, 26, 29, 30, 31a, 32,** and **33a.**[3] The known compound **27** was isolated as a pure sample and showed NMR spectra identical to those reported.[19] ^{11}B NMR was obtained at 128.32 MHz, and the chemical shifts are in δ units relative to $Et_2O{-}BF_3$ (0.0 ppm in $CDCl_3$).

Synthesis of $NaB(OMe)_4$. A solution of $NaBH_4$ (500 mg) in MeOH (25 mL) was refluxed for 30 min. The solvent was removed, giving 2.1 g of **1** (100% yield): white solid; ^{11}B NMR (128.32 MHz, CD_3OD) δ 3.02 (s).

Synthesis of Michael Donor 14. A sample of (*Z*)-$BrCH_2CH{=}CHCH_2OCO_2Et$[20] (500 mg, 2.2 mmol) was added to a mixture of NaH (106 mg, 4.4 mmol) and dimethyl malonate (871 mg, 6.6 mmol) in DMF (40 mL). This solution was stirred at room temperature for 4 h and then diluted with Et_2O, washed with 2 N HCl, and dried with anhydrous Na_2SO_4, and the solvent was removed. The residue was submitted to flash chromatography (7:3 hexane/EtOAc) to give **14** (442 mg, 72%) as a colorless oil; ^1H NMR (500 MHz, $CDCl_3$) δ 5.60−5.54 (m, 1H), 5.52−5.47 (m, 1H), 4.59 (d, $J = 5.5$ Hz, 2H), 4.08 (q, $J = 7.1$ Hz, 2H), 3.64 (s, 6H), 3.34 (t, $J = 7.4$ Hz, 1H), 2.61 (t, $J = 6.8$ Hz, 2H), 1.19 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$; DEPT) δ 169.2 (C), 155.3 (C), 130.3 (CH), 126.5 (CH), 64.2 (CH_2), 63.3 (CH_2), 52.8 (CH_3), 51.5 (CH), 27.2 (CH_2), 14.46 (CH_3); FABHRMS calcd for $C_{12}H_{18}O_7Na$ *m/z* 297.0950, found *m/z* 297.0949.

Model Procedure for the $NaB(OMe)_4$-Catalyzed Michael Reaction. The Michael acceptor (1.0 or 2.0 mmol, see Table 1) was added to a mixture of Michael donor (1.0 mmol) and $NaB(OMe)_4$ (0.03 mmol, 3 mol %) in MeCN (3 mL) at room temperature. The resulting solution was stirred at room temperature for 3−24 h (see Table 1). The solvent was removed, and the residue was chromatographed (hexane/EtOAc mixtures) to give adducts **4**, **19**−**35** at the yields indicated in eq 1 and Table 1.

Compound 28: colorless oil; ^1H NMR (500 MHz, $CDCl_3$) δ 3.74 (s, 3H), 3.72 (s, 3H), 2.71 (dd, $J = 7.4, 4.2$ Hz, 1H), 2.56 (dt, $J = 17.8, 7.8$ Hz, 1H), 2.40 (dt, $J = 17.8, 7.6$ Hz, 1H), 2.26 (t, $J = 7.5$ Hz, 2H), 2.22 (dd, $J = 14.9, 4.2$ Hz, 1H), 2.13 (s, 3H), 1.98 (dd, $J = 14.9, 7.4$ Hz, 1H), 1.27 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$; DEPT) δ 207.2 (C), 171.6 (C), 171.5 (C), 60.0 (CH), 58.1 (C), 56.0 (C), 52.9 (CH_3), 52.8 (CH_3), 38.9 (CH_2), 33.4 (CH_2), 30.1 (CH_3), 27.5 (CH_2), 24.9 (CH_3), 18.9 (CH_3). FABHRMS calcd for $C_{14}H_{22}O_6Na$ *m/z* 309.1314, found *m/z* 309.1308.

Compound 31b. Minor diastereomer **31b** was obtained as a 4:1 mixture with the known polyketone **32** and showed the following NMR data: ^1H NMR (300 MHz, $CDCl_3$) δ 2.70 (dd, $J = 12.9, 3.7$ Hz, 1H), 2.60 (ddd, $J = 13.9, 3.1, 2.7$ Hz, 1H), 2.47−2.38 (m, 1H), 2.31 (s, 3H), 2.16 (s, 3H), 2.14 (s, 3H), 1.85−1.65 (m, 2H), 1.46 (td, $J = 14.0$, 4.1 Hz, 1H), 1.18 (s, 3H), 1.26−1.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ 211.5 (C), 206.2 (C), 205.1 (C), 71.7 (C), 67.9 (C), 55.4 (CH), 38.5 (CH_2), 31.7 (CH_3), 29.2 (CH_2), 27.1 (CH_2), 26.4 (CH_3), 25.9 (CH_3), 21.7 (CH_3); FABHRMS calcd for $C_{13}H_{20}O_4Na$ *m/z* 263.1259, found *m/z* 263.1258.

Compound 33b. Minor diastereomer **33b** was obtained as a 1:1 mixture of epimers at C-1; colorless oil; one diastereomer: ^1H NMR (500 MHz, $CDCl_3$) δ 3.77 (s, 3H), 2.68 (dd, $J = 9.0, 3.8$ Hz, 1H),

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(10) $NaBH_4$ shows a quintuplet signal at −35.6 ppm (DMSO-d_6).

(11) We also carried out the same reaction using other solvents, but the yields were lower. See Supporting Information.

(12) Although significant differences in absolute values between p*K*a values measured in DMSO or acetonitile are expected, the relative acidities do not differ greatly. For that discussion and p*K*a values of organic compounds in DMSO at 25 °C, see: Bordwell, F. G. *Acc. Chem. Res.* **1988**, *21*, 456−463.

(13) A $\log K = 5.62$ was determined for the equilibrium between $MeO^- + B(OMe)_3$ and $[B(OMe)_4]^-$ in MeOH: (a) Gut, R. *Helv. Chim. Acta* **1964**, *47*, 2262−2278. (b) Hutton, W. C.; Crowell, T. I. *J. Am. Chem. Soc.* **1978**, *100*, 6904−6907.

(14) $NaBF_4$ is unable to promote the Michael reaction, ruling out the role of sodium cation as Lewis acid.

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2.49 (dd, $J = 3.8, 2.5$ Hz, 1H), 2.45 (t, $J = 2.9$ Hz, 1H), 2.29 (m, 1H), 2.25 (s, 3H), 2.14 (s, 3H), 1.75 (t, $J = 13.5$ Hz, 1H), 1.69−1.64 (m, 1H), 1.44−1.38 (m, 1H), 1.16 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$; DEPT) δ 211.8 (C), 204.6 (C), 172.0 (C), 71.8 (C), 61.4 (C), 55.9 (CH), 53.0 (CH_3), 38.8 (CH_2), 31.6 (CH_3), 30.2 (CH_2), 27.9 (CH_2), 26.3 (CH_3), 21.7 (CH_3). Another diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.70 (s, 3H), 2.69 (dd, $J = 8.5, 4.0$ Hz, 1H), 2.52 (dd, $J = 4.0, 2.5$ Hz, 1H), 2.42 (t, $J = 2.9$ Hz, 1H), 2.32 (m, 1H), 2.24 (s, 3H), 2.17 (s, 3H), 1.82 (t, $J = 13.4$ Hz, 1H), 1.71−1.68 (m, 1H), 1.61−1.53 (m, 1H), 1.18 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$; DEPT) δ 211.7 (C), 204.2 (C), 172.3 (C), 71.6 (C), 59.7 (C), 55.4 (CH), 53.0 (CH_3), 37.9 (CH_2), 30.2 (CH_3), 29.7 (CH_2), 27.8 (CH_2), 25.9 (CH_3), 22.1 (CH_3); FABHRMS calcd for $C_{13}H_{20}O_5Na$ m/z 279.1208, found m/z 279.1208.

Compound 34: colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.62−5.56 (m, 1H), 5.44−5.39 (m, 1H), 4.55 (d, $J = 6.8$ Hz, 2H), 4.08 (q, $J = 7.1$ Hz, 2H), 3.61 (s, 6H), 2.60 (d, $J = 7.5$ Hz, 2H), 2.36 (t, $J = 8.2$ Hz, 2H), 2.04 (t, $J = 8.2$ Hz, 2H), 2.02 (s, 3H), 1.19 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$; DEPT) δ 207.1 (C), 171.3 (C), 155.2 (C), 128.5 (CH), 127.2 (CH), 64.2 (CH_2), 63.1 (CH_2), 56.8 (C), 52.7 (CH_3), 38.7 (CH_2), 31.9 (CH_2), 30.0 (CH_3), 26.9 (CH_2), 14.4 (CH_3); FABHRMS calcd for $C_{16}H_{24}O_8$-Na m/z 367.1368, found m/z 367.1363.

Compound 35: colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.70 (dt, $J = 15.0, 5.7$ Hz, 1H), 5.52 (dt, $J = 15.0, 5.7$ Hz, 1H), 4.06 (d, $J = 5.8$ Hz, 2H), 3.70 (s, 6H), 2.62 (d, $J = 7.2$ Hz, 2H), 2.44 (t, $J = 8.0$ Hz, 2H), 2.12 (s, 3H), 2.11 (t, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$; DEPT) δ 207.4 (C), 171.5 (C), 134.2 (CH), 125.7 (CH), 63.3 (CH_2), 57.1 (C), 52.6 (CH_3), 38.7 (CH_2), 36.8 (CH_2), 30.1 (CH_3), 26.8 (CH_2); FABHRMS calcd for $C_{13}H_{20}O_6$-Na m/z 295.1157, found m/z 295.1159.

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Supporting Information Available: Additional experiments and characterization data. This material is available free of charge via the Internet at http://pubs.acs.org.

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