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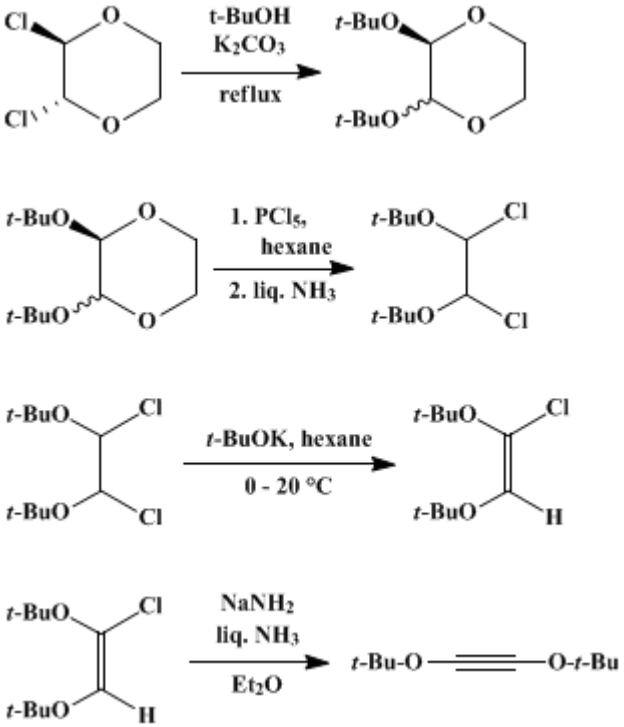
DIALKOXYACETYLENES: DI-*tert*-BUTOXYETHYNE, A VALUABLE SYNTHETIC INTERMEDIATE

[Propane, 2,2'-j[1,2-ethynediylbis(oxy)]bis[2-methyl-]



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1. Procedure

A. (1) *Preparation of trans-2,3-dichloro-1,4-dioxane.* (See (Note 1).) To a 2-L, three-necked, round-bottomed flask, equipped with two inlet tubes (with sintered-glass diffusers at the end) connected to a chlorine cylinder, and a reflux condenser connected to an outlet tube immersed in a potassium hydroxide solution, are added 1200 g (13.64 mol) of anhydrous dioxane (*free of peroxides!*) and 8 g (0.03 mol) of iodine. A stream of chlorine is passed through the dioxane/iodine solution heated at 90°C, and the reaction is monitored by NMR spectroscopy. After 9 hr, the conversion is 50% complete (Note 2); after 33 hr, about 90% complete. At this point, the stream of chlorine is interrupted. *Reinitiation of the chlorine stream after some hours (next morning, for example) may be dangerous because it was observed in one case the mixture inflamed spontaneously!* The reaction mixture is allowed to cool to room temperature, 500 mL of ether is added, and the solution is washed with aqueous sodium thiosulfate solution. The organic layer is separated, dried over sodium sulfate, the ether is evaporated under reduced pressure, and the residue is distilled through a 20-cm Vigreux column, to yield 1200–1300 g of *trans*-2,3-dichloro-1,4-dioxane, bp 89°C/16 mm (lit.[1] bp 82.5°C/14 mm; mp 31°C) (Note 3).

(2) *2,3-Di-tert-butoxy-1,4-dioxane.* To a 2-L, three-necked, round-bottomed flask, equipped with a mechanical stirrer, a reflux condenser protected from moisture by a drying tube, and an inlet tube for dry nitrogen, are added 103.6 g (0.66 mol) of *trans*-2,3-dichloro-1,4-dioxane, 979.1 g (13.23 mol) of anhydrous *tert*-butyl alcohol (distilled from CaH_2), and 365.1 g (2.64 mol) of potassium carbonate (ground with a mortar and pestle and activated at 250°C for 3 hr) (Note 4). The mixture is stirred vigorously and heated under reflux for 24–30 hr, the progress of the reaction being monitored by ^1H

NMR spectroscopy. Once the singlet at δ 5.95, corresponding to the methine protons of the starting material, has completely disappeared, the reaction mixture is allowed to cool to room temperature, and poured into 500 mL of ether, and enough water (750–850 mL) is added to dissolve all of the inorganic salts. The organic layer is separated, and the aqueous layer is extracted with two 200-mL portions of ether. The combined ether extracts are dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure to give 128–147 g of an oily residue (Note 5). Hexane (250 mL) is added and the solution is allowed to stand in a refrigerator. The resulting crystals are separated by suction filtration and washed thoroughly with 150–250 mL of hexane. The residue of 3–6 g of crystalline product, mp 106–107°C, which remains insoluble, is identified as *trans*-2-*tert*-butoxy-3-hydroxy-1,4-dioxane (Note 6). The filtrate (which contains approximately a 25 : 75 mixture of *cis* and *trans* isomers) is evaporated under reduced pressure to one-half its volume and is cooled to 0°C. Massive crystals appear, which are collected by suction filtration. The crystallization process is repeated once more to give 40–60 g of *trans*-2,3-di-*tert*-butoxy-1,4-dioxane, mp 64–65°C. The hexane solution is evaporated under reduced pressure and the oily residue distilled at 50–57°C/0.25 mm to give 54–84 g of a mixture of *cis*- and *trans*-2,3-di-*tert*-butoxy-1,4-dioxane (115–124 g combined; 75–81% yield) (Note 7).

B. *1,2-Di-tert-butoxy-1,2-dichloroethane.* To a 250-mL, round-bottomed flask, equipped with a pressure-equalizing dropping funnel protected from moisture by a drying tube, and a magnetic stirring bar, are added 43.2 g (0.21 mol) of phosphorus pentachloride and 50 mL of hexane, and the flask is cooled with an ice–salt bath. While the solution is stirred, a solution of 30 g (0.13 mol) of 2,3-di-*tert*-butoxy-1,4-dioxane in 100 mL of hexane is added dropwise. After the addition is complete, the cooling bath is removed and the mixture is stirred at room temperature for 90–180 min until no starting material is observed in the NMR spectrum of a sample (Note 8); the reaction mixture is then filtered through a sintered-glass filter to remove excess phosphorus pentachloride. The resulting hexane solution (≈ 200 mL), which contains 1,2-di-*tert*-butoxy-1,2-dichloroethane, 2-chloroethyl dichlorophosphate, 1,2-dichloroethane, phosphorus oxychloride, and traces of phosphorus pentachloride, is transferred into a 1-L, three-necked, round-bottomed flask, equipped with a magnetic stirrer, a dry-ice condenser protected from moisture by a potassium hydroxide tube, and a short inlet tube connected to an ammonia cylinder. The reaction flask is cooled with a dry ice–acetone bath and, while the solution is stirred (Note 9), a fast stream of gaseous ammonia is introduced. A vigorous reaction takes place and a copious white precipitate forms. The stream of gaseous ammonia is continued for 15–60 min (Note 10). The cooling bath and condenser are removed, the reaction flask is connected to an ordinary aspirator line through a potassium hydroxide drying trap, and the ammonia is evaporated under aspirator vacuum with efficient stirring. The ammonia-free solution is filtered through a sintered-glass filter and the precipitate is washed with hexane. The resulting hexane solution (400–500 mL), which contains 25–26 g (0.12 mol) of pure 1,2-di-*tert*-butoxy-1,2-dichloroethane, is suitable for the next operation (yields, calculated from an aliquot, are 95–97%) (Note 11).

C. *(E)-1,2-Di-tert-butoxy-1-chloroethene.* The solution prepared above is placed in a 1-L, round-bottomed flask, equipped with a magnetic stirring bar and a Liebig condenser (protected from moisture by a drying tube) and cooled with an ice bath; 28.9 g (0.258 mol) of solid potassium *tert*-butoxide is added in small portions through the condenser over a 30-min period. After addition, the cooling bath is removed and stirring is continued for 90–120 min until no more starting material is detected in the ^{1}H NMR spectrum of a sample; enough ice water is then added to just dissolve all the inorganic salts. The organic layer is separated and the aqueous layer is extracted with hexane (2 × 100 mL). The combined hexane extracts are dried over anhydrous sodium sulfate, filtered, and concentrated at aspirator vacuum. The residue is distilled at 40°C/0.2 mm, with the collection flask at −78°C, to give a center cut of (*E*)-1,2-di-*tert*-butoxy-1-chloroethene (15.4–17.0 g, 58–63% yield from 2,3-di-*tert*-butoxy-1,4-dioxane) (Note 12) and (Note 13).

D. *Di-tert-butoxyethyne.* In a 2-L, three-necked, round-bottomed flask, equipped with a magnetic stirring bar (Note 14), a dry-ice condenser protected from moisture by a potassium hydroxide tube, and a pressure-equalizing dropping funnel, 0.5 mol of sodium amide is prepared in 500 mL of liquid ammonia (Note 15), and 20 g (0.0968 mol) of (*E*)-1,2-di-*tert*-butoxy-1-chloroethene dissolved in 150 mL of anhydrous ether is added in a 5-min period with efficient stirring. After the addition, stirring is continued for 80 min. The reaction mixture is diluted with 200 mL of cold pentane (−20 to −30°C), and 400 mL of cold water is added very cautiously. The organic layer is washed with 50 mL of a 0.1 *M*

buffered phosphate solution (NaH_2PO_4/Na_2HPO_4), dried over anhydrous sodium sulfate (Note 16), filtered, and concentrated at aspirator vacuum without heating to give 10–12 g (78–86% yield) of di-*tert*-butoxyethyne as a pale-yellow oil. The product is sufficiently pure for further reactions (Note 17), but it may be distilled at 30°C/0.05 mm; freezing point 8.5°C.

2. Notes

1. This procedure was reported by J. J. Kucera and D. C. Carpenter.[1,2]
2. These data were obtained by the checkers. The submitters report conversion of 76–80% after only 9 hr. It seems likely that the rate of the reaction may be sensitive to the dimensions and mechanical features of the chlorine introduction system, and/or an induction period. It is easy and important to monitor the process.
3. *trans*-2,3-Dichloro-1,4-dioxane has the following spectra: IR (CCl_4) cm^{-1}: 2990, 2940, 2885, 1455, 1385, 1375, 1337, 1160, 1115, 1032, 900, 875, 670; ^1H NMR (CCl_4)[2b] δ: 3.40–4.57 (AA'BB', 4 H, CH_2), 5.95 (s, 2 H, ClCHO).
4. The yields of this reaction are very sensitive to the presence of traces of moisture and to the ratio of reagents. If one works without a nitrogen atmosphere and with *tert*-butyl alcohol that has not been previously dried over calcium hydride, with a 1 : 10 ratio of dichloro derivative/alcohol, the yields drop to 65%.
5. *Caution should be exercised in evaporation of the ether, as the di-tert-butoxy compounds are appreciably volatile at reduced pressure. If a rotary evaporator is used for the concentration, the water bath should be kept at or below room temperature, and the residue should not be pumped after it is clear that the bulk of the ether has been evaporated.*
The submitters report ca. 130 g of oily residue at this stage. Treatment of the oily residue with 105 mL of ether leads to partial crystallization. During washing of the crystals in a Büchner filter with more ether, an almost complete solubilization takes place, but eventually 0.1–0.5 g of *cis*-2,3,7,10-tetraoxabicyclo[4.4.0]decane remains as an insoluble residue. This compound was prepared for the first time in 1931;[3] the *cis* configuration was established only in 1966.[4] It has the following properties: mp 136°C; IR (CCl_4) cm^{-1}: 2980, 2955, 2930, 2910, 2875, 1460, 1350, 1285, 1260, 1250, 1150, 1140, 1095, 1080, 1025, 910, 870, 780; ^1H NMR (CCl_4) δ: 3.30–4.20 (AA'BB', 8 H CH_2), 4.60 (s, 2 H, OCHO).
6. The crystalline trans isomer epimerizes in chloroform solution to give a nearly 70 : 30 mixture of *cis* and *trans* isomers. The *trans* isomer, mp 106–107°C, shows the following spectroscopic properties: IR (KBr) cm^{-1}: 3450, 2980, 2935, 2890, 1445, 1390, 1370, 1335, 1280, 1260, 1200, 1135, 1105, 1060, 1045, 1035, 1020, 910, 855, 780; ^1H NMR (CCl_4) δ: 1.27 (s, 9 H, CH_3), 3.33–4.13 (ABCD + OH, 4 H + 1 H) 4.57 (br, 2 H, OCHO). Anal. calcd. for $C_8H_{16}O_4$: C, 54.53; H, 9.15. Found: C, 54.53; H, 9.29.
7. *trans*-2,3-di-*tert*-butoxy-1,4-dioxane has the following spectra: IR (CCl_4) cm^{-1}: 2975, 2930, 1390, 1367, 1190, 1145, 1100, 1060, 1040, 857; ^1H NMR (CCl_4) δ: 1.19 (s, 18 H, CH_3), 3.05–4.20 (m, AA'BB', 4 H, CH_2), 4.30 (s, 2 H, OCHO). *cis*- + *trans*-2,3-Di-*tert*-butoxy-1,4-dioxane have the following additional signals: IR (CCl_4) cm^{-1}: 1170, 1130, 1120, 1080, 1020, 1000, 960, 879; ^1H NMR (CCl_4) δ: 4.43 (s, 2 H, OCHO, *cis*).
8. The peaks corresponding to 2,3-di-*tert*-butoxy-1,4-dioxane overlap with those of the methylene protons of 2-chloroethyl dichlorophosphate, a by-product from the reaction, but the absence of the acetal protons of the starting material is clear from the symmetry of the multiplet.
9. The solution is thick at −78°C. Dilution with additional hexane may be necessary.
10. The end of the reaction can be easily detected because, when all of the 2-chloroethyl dichlorophosphate has been destroyed, the reaction mixture *does not crackle* any more when condensed ammonia drops on the stirred mixture, or, much more easily, when the reaction mixture becomes basic to pH paper.
11. The crude reaction mixture is, in fact, an approximately 30 : 70 mixture of *dl*- and *meso*-1,2-di-*tert*-butoxy-1,2-dichloroethane. Although the ^1H NMR spectrum at 60 MHz (CCl_4) shows only one singlet at 5.6 ppm, the 200-MHz spectrum ($CDCl_3$) shows two sharp singlets separated by 1.8 Hz. The pure meso compound could be isolated by crystallization and purified by sublimation at 40°C/0.05 mm; mp 77–78° (dec). The spectra are as follows: IR (CCl_4) cm^{-1}: 2975, 2925, 1470, 1458, 1390, 1368, 1310, 1250, 1180, 1130, 1025, 850, 650; ^1H NMR at 200 MHz ($CDCl_3$) δ: 1.36 (s, 9 H, CH_3), 5.73 (s, 1 H, OCHCl).
12. In later fractions, small amounts (0.2–0.5%) of (*Z*)-1,2-di-*tert*-butoxy-1-chloroethane have been detected: ^1H NMR (CCl_4) δ: 1.25 (s, 9 H, CH_3), 1.33 (s, 9, H, CH_3), 6.03 (s, 1 H, = CH).

13. (*E*)-1,2-Di-*tert*-butoxy-1-chloroethene has the following properties: n_D 1.4410–1.4415; UV (cylohexane): 217.7 nm (log ε = 3.7); IR (CCl_4) cm^{-1}: 2972, 1670, 1470, 1390, 1366, 1290, 1260, 1240, 1180, 1140, 1070, 1025, 935; ^1H NMR (CCl_4) δ: 1.26 (s, 9 H, CH_3), 1.33 (s, 9 H, CH_3), 5.91 (s, 1 H, = CH).
14. The stirring bar must be glass-covered, since sodium in ammonia solution attacks Teflon.
15. The method used for the preparation of sodium amide is a modification of the procedure described by Nieuwland et al.[5] In a 3-L, three-necked, round-bottomed flask, equipped with a magnetic stirring bar (Note 14), a dry-ice condenser protected from moisture by a potassium hydroxide tube, and an inlet tube connected to the ammonia cylinder, is condensed 500 mL of liquid ammonia. A slow stream of dry oxygen is initiated through the inlet tube and 11.5 g (0.5 mol) of sodium in small pieces is slowly introduced. The addition of sodium requires 4–5 hr, since the blue color must be discharged before each new addition of sodium. In this way, a completely white suspension of sodium amide is obtained, which allows the formation of crude di-*tert*-butoxyethyne, free from any iron impurities.
16. It is best to minimize exposure of di-*tert*-butoxyethyne to light.
17. Eventually, if a more concentrated solution of (*E*)-1,2-di-*tert*-butoxy-1-chloroethene in ether is used, the formation of 1,2,3-tri-*tert*-butoxy-3-cyano-1-propene [^1H NMR (CCl_4) δ: 6.05 (s, 1 H), 4.98 (s, 1 H), 1.28 (br, 27 H)] and 1,2,3-tri-*tert*-butoxy-1-cyano-1-propene [^1H NMR (CCl_4) δ: 4.03 (s, 2 H), 1.28 (b, 27 H)] is observed. The by-products may be eliminated by column chromatography on neutral alumina (40 g, 100–125 mesh, activity 1), using a column refrigerated at 0°C and protected from the light, and eluting with pentane under nitrogen pressure. From the first 750 mL of eluant, 9–12 g of pure di-*tert*-butoxyethyne is obtained.
18. Di-*tert*-butoxyethyne has the following properties: n_D^{25} 1.4365; IR (CCl_4) cm^{-1}: 2972, 2922, 1470, 1450, 1390, 1367, 1301, 1263, 1245, 1150, 825; ^1H NMR (CCl_4) δ: 1.31 (s, CH_3).

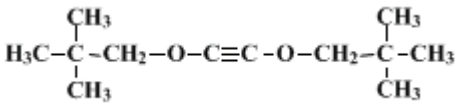
3. Discussion

The present procedure for the preparation of di-*tert*-butoxyethyne is an improvement of a method previously reported by the submitters,[6] who have also reported the preparation from glyoxal via 1,2-dichloro-1,2-dimethoxyethane.[6]

Although acetylenic diethers are thermodynamically stable compounds, they show a high kinetic instability that induces polymerization even at low temperatures.[7]

Different acetylenic diethers have been prepared, either from glyoxal (method B) or from dioxane (method A); their stability correlates well, in a qualitative way, with Charton's ν steric parameter,[8] based on effective Van der Waals radii for the corresponding alkoxy groups (Table I).

TABLE I
ACETYLENIC DIETHERS

RO–C≡C–OR -OR	Method	ν	$t_{1/2}$ Order of magnitude (by NMR)
$H_3CO-C\equiv C-OCH_3$ $-OCH_3$	B[9]	0.38	Seconds (0°C, soln.)
$C_2H_5O-C\equiv C-OC_2H_5$ $-OC_2H_5$	B_7	0.48	Seconds (0°C, soln.)
	A[10]	0.70	Minutes (RT, [a]soln.)

$-OCH_2C(CH_3)_3$			
$(CH_3)_2HCO-C\equiv C-OCH(CH_3)_2$	A,B[6]	0.75	Minutes (RT, soln.)
$-OCH(CH_3)_2$			
$(CH_3)_3CO-C\equiv C-OC(CH_3)_3$	A,B[6]	1.22	Days (RT, neat)
$-OC(CH_3)_3$			

[a] Room temperature

.

As shown above, di-*tert*-butoxyethyne is the only acetylenic diether prepared so far whose stability allows its use as a synthetic intermediate. It has been used in the synthesis of all the members of the series of monocyclic oxocarbons (deltic, squaric, croconic, and rhodizonic acids),[9 10 11 12] as well as in the synthesis of semisquaric acid, the parent compound of the natural mycotoxins, *moniliformins*.[10]

Di-*tert*-butoxyethyne, like other acetylenic ethers having hydrogen atoms β to oxygen, is thermally unstable, and when a benzene solution is heated under reflux, elimination occurs to give isobutene and *tert*-butoxyketene. *tert*-Butoxyketene then reacts with the parent acetylene to afford 2,3,4-tri-*tert*-butoxycyclobutenone, which is the precursor of squaric and semisquaric acid.[9,10] This thermal instability prevents the use of di-*tert*-butoxyethyne in those reactions that proceed at temperatures higher than 40–50°C.

On the other hand, di-*tert*-butoxyethyne is prone to undergo a variety of reactions with transition-metal complexes [$PdCl_2CH_3CN$, $Co_2(CO)_8$, $CpCo(CO)_2$, $Ni(CO)_4$], to afford new, transient, intermediate complexes which are the actual precursors of oxocarbons.[13]

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Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

dl- and meso-1,2-di-tert-butoxy-1,2-dichloroethane

Propane, 2,2'-j[1,2-ethynediylbis(oxy)]bis[2-methyl-

ACETAL (105-57-7)

potassium carbonate (584-08-7)

acetylene (74-86-2)

ammonia (7664-41-7)

Benzene (71-43-2)

ether (60-29-7)

hydrogen (1333-74-0)

glyoxal (107-22-2)

phosphorus pentachloride (10026-13-8)

chloroform (67-66-3)

iron (7439-89-6)

sodium sulfate (7757-82-6)

oxygen (7782-44-7)

sodium thiosulfate (7772-98-7)

1,2-dichloroethane (107-06-2)

nitrogen (7727-37-9)

iodine (7553-56-2)

methine (7782-42-5)

Phosphorus Oxychloride (21295-50-1)

chlorine (7782-50-5)

potassium hydroxide (1310-58-3)

sodium (13966-32-0)

methylene (2465-56-7)

Pentane (109-66-0)

dioxane (5703-46-8)

isobutene (9003-27-4)

sodium amide (7782-92-5)

hexane (110-54-3)

tert-butyl alcohol (75-65-0)

calcium hydride (7789-78-8)

phosphate

trans-2,3-Dichloro-1,4-dioxane (3883-43-0)

2-chloroethyl dichlorophosphate (1455-05-6)

1,2-dichloro-1,2-dimethoxyethane

potassium tert-butoxide (865-47-4)

2,3-Di-tert-butoxy-1,4-dioxane,
cis- and trans-2,3-di-tert-butoxy-1,4-dioxane (68470-79-1)

1,2-Di-tert-butoxy-1,2-dichloroethane (68470-81-5)

(E)-1,2-Di-tert-butoxy-1-chloroethene (70525-93-8)

cis-2,3,7,10-tetraoxabicyclo[4.4.0]decane

(Z)-1,2-di-tert-butoxy-1-chloroethane

1,2,3-tri-tert-butoxy-3-cyano-1-propene

1,2,3-tri-tert-butoxy-1-cyano-1-propene

tert-butoxyketene

2,3,4-tri-tert-butoxycyclobutenone

Di-tert-butoxyethyne (66478-63-5)

trans-2-tert-butoxy-3-hydroxy-1,4-dioxane

trans-2,3-di-tert-butoxy-1,4-dioxane