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REVIEW

Advancements in tetronic acid chemistry. Part 1: Synthesis and reactions

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KEYWORDS

Tetronic acid;
Synthetic routes;
Chemical reactivity;
Tautomeric structure;
Reactions

Abstract The preparation and the properties of the elusive tetronic acid are reviewed, including its synthesis, chemical reactivity and reactions.

Contents

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1. Introduction

Tetronic acids, 4-hydroxy-2(5H)-furanones, were discovered in 1896 by Wolffe and Schwabe (1896). They are a structural fragment of a series of natural compounds, such as acetogenins, muconolactones, leptosfaerin, and strigol (Cain et al., 1989; De Guzman and Schnutz, 1990; Iida et al., 1997; Miller and Hegedus, 1993; Miller et al., 1998; Ribbons and Sutherland, 1994; Rupprecht et al., 1990; Schiehser et al., 1986; Tejedor and García-Tellado, 2004; Zografos and Georgiadis, 2006). They represent an interesting template for medicinal chemistry because of their antibiotic (Brodersen and Kjaer, 1946; Capon and MacLeod, 1987; Haynes and Plimmer, 1960; Ley et al., 1991; Matsumoto et al., 1990; Vanwagenen and Cardellina, 1986; Vieweg et al., 2014), HIV-1 protease inhibitors (Roggo et al., 1994), anticoagulant (Rehse and Emisch, 1983; Rehse and Rothe, 1982; Rehse et al., 1978; Witiak et al., 1982), antiepileptic (Zhang et al., 1992), antibacterial (Murray et al., 2014), antifungal (Luk and Readshaw, 1991; Vishwakarma et al., 1987; Hu et al., 2014), insecticidal (Ibi et al., 1979), analgesic (Dal Pozzo et al., 1974) and anti-inflammatory activities (Foden et al., 1975). Recently, these compounds have also been reported as anticancer agents (Andreoli et al., 2014; Han et al., 2014; kamal et al., 2014a, 2014b).

In continuation of our studies in exploring the utilization of cyclic 1,3-dicarbonyl compounds as versatile precursors for synthesis of organic compounds (Abdou et al., 2015a, 2015b; Abdou, 2014a, 2014b, 2014c, 2013; Metwally et al., 2013a, 2013b, 2012a, 2012b, 2012c, 2012d, 2012e). The aim of this review was to give an account of the principal literature on methods of synthesis and the reactivity of tetronic acid (without any substituents attached). The discussion is supported by numerous lucid diagrams and the extensive reaction schemes are supported by relevant and up-to-date references from the original literature.

2. Tautomeric structure(s)

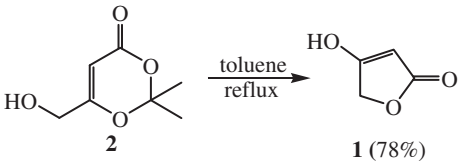
Tetronic acid can exist in either the keto- or enol-form (Fig. 1). These four possible prototropic transformations have been intensively examined by various chemical reactivity, spectral, thermochemical, and computational methods (Ballantine et al., 1968; Duncanson, 1953; Edsall and Sagall, 1943; Haynes et al., 1968; Jurd, 1996; Martoglio and Katon, 1993; Pollet et al., 1984; Zimmer et al., 1978).

3. Synthesis

The syntheses of tetronic acid are reported throughout the literature (Allan et al., 1983; Bloomer and Kappler, 1976; Greenhill et al., 1975; Greenhill and Tomassini, 1974; Haynes and Plimmer, 1960; Pollet and Gelin, 1978; Schmidt and Zimmer, 1981; Svendsen and Boll, 1973; Zimmer et al., 1978), although very little has been done to concentrate the wide range of syntheses into a single report.

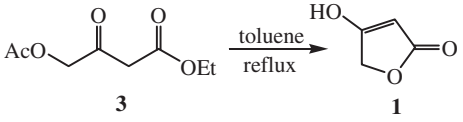
3.1. Using 1, 3-dioxin-4-one

Sato et al. (1990) disclosed a simple and inexpensive synthesis of tetronic acid **1** in 78% yield *via* refluxing of 6-(hydroxymethyl)-dioxin-4-one **2** in toluene (Scheme 1).



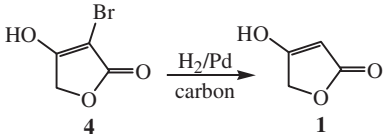
2 **1** (78%)

Scheme 1



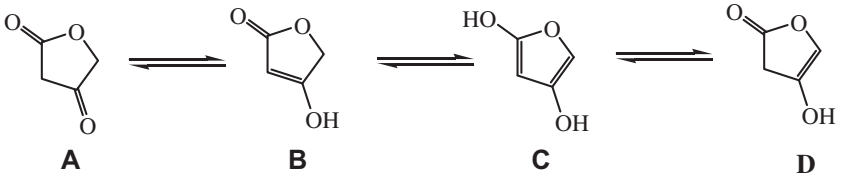
3 **1**

Scheme 2



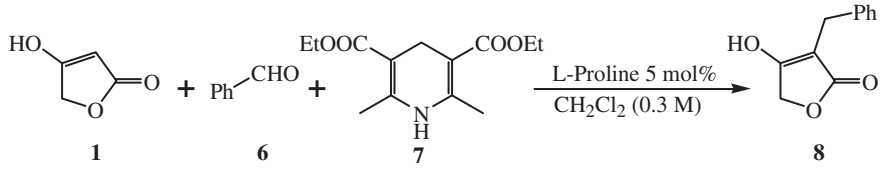
4 **1**

Scheme 3

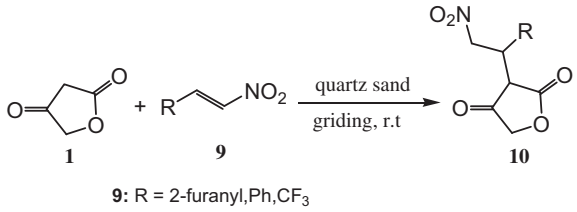


A **B** **C** **D**

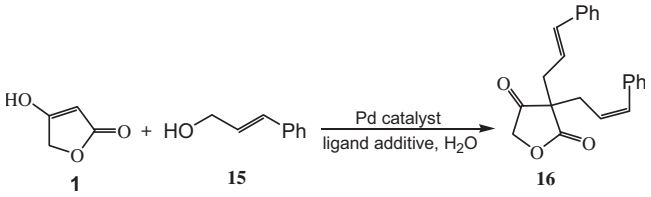
Figure 1 Possible tautomeric structures of tetronic acid **1** (A–D).



Scheme 4



Scheme 5



Scheme 7

3.2. Using lactonization of γ-acetoxy-β-keto esters

Gelin and Pallet (1980) reported that the desired tetronic acid **1** could be obtained by lactonization of γ-acetoxy-β-keto ester **3** in refluxing toluene (Scheme 2).

3.3. Debromination of α-bromotetronic

A catalytic debromination of α-bromotetronic **4** with palladium on carbon, under approximately 40 lbs of hydrogen pressure, results in 90% yield of **1** (Schmidt and Zimmer, 1981) (Scheme 3).

4. Chemical reactions

In this section, all the reactions with participation of tetronic acid on the basis of the new bond formed.

4.1. Reactions involving carbon–carbon bond formation

4.1.1. C–C bond formation reactions

4.1.1.1. C_3-alkylation. Ramachary and Kishor (2010) observed that the reaction of tetronic acid **1** with benzaldehyde **6** and diethyl 2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate **7** under proline catalysis in dichloromethane gave 3-benzyl-tetronic acid **8** (Scheme 4).

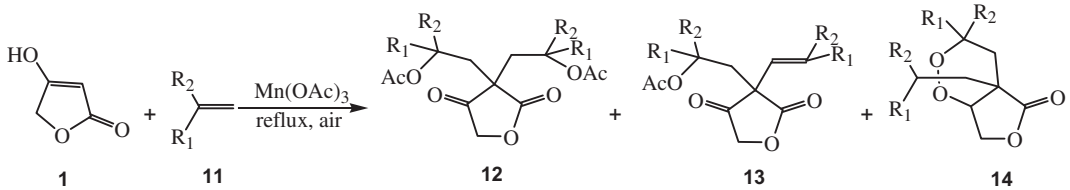
Michael addition reactions of **1** to β-nitroalkenes **9** afforded 3-(1-substituted-2-nitro-ethyl)-furan-2,4-diones **10.** This reaction requires a basic catalyst in organic solvents, as well as long reaction times, which may lead to environmentally hazardous residues and undesirable by-products (Iwata et al., 1993). Recently, an environmentally benign, fast and convenient protocol for this reaction by a grinding method under catalyst and solvent-free conditions was reported (Xie et al., 2012) (Scheme 5).

The double alkylation of tetronic acid **1** with 1,1-disubstituted alkenes **11** in the presence of manganese(III) acetate in acetic acid at room temperature afforded the diethyl- and/or ethenyl-ethyl-substituted tetronic acids **12** and/or **13** along with peroxypropellane 14 (Haque and Nishino, 2011) (Scheme 6).

4.1.1.2. C_3-allylation reaction. The allylation of **1** is an important strategy for the formation of C–C bonds in organic synthesis. Activator-free and one-pot C-allylation of **1** by simple palladium catalyst in water using cinnamyl alcohol **15** and heating gave the corresponding C-allylated product **16** (Gan et al., 2008; Shue and Yang, 2012) (Scheme 7).

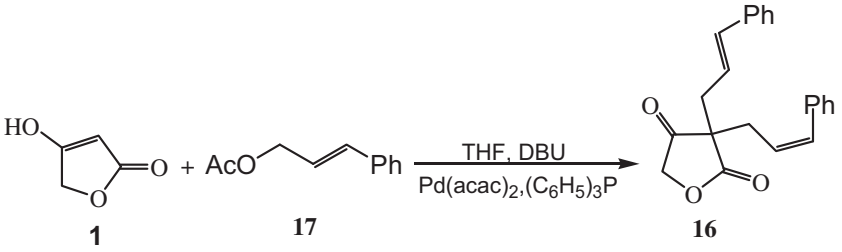
Prat et al. (1988) reported that tetronic acid **1** is efficiently alkylated with cinnamyl acetate **17** to afford **16** (38%) using 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), palladium(II) 2,4-pentanedionate, triphenylphosphine as catalyst in tetrahydrofuran (Scheme 8).

The above methodology (Moreno-Mañas et al., 1988; Prat et al., 1988) was successfully applied for the alkylation by using 2-cyclohexenyl acetate **18**. This reaction proceeds smoothly to give the corresponding monoalkylated lactone **19** (57%) although minor amounts of the dialkylated butanolide **20** were also isolated (Scheme 9).

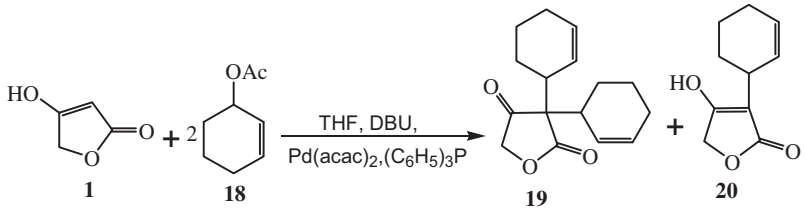


R_3= H, R_1=R_2= 4-ClC_6H_4, Ph

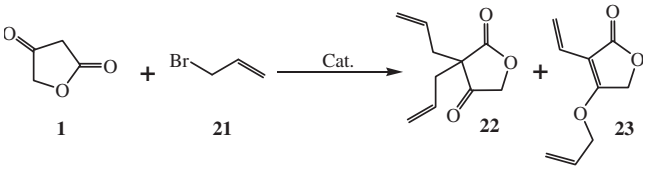
Scheme 6



Scheme 8



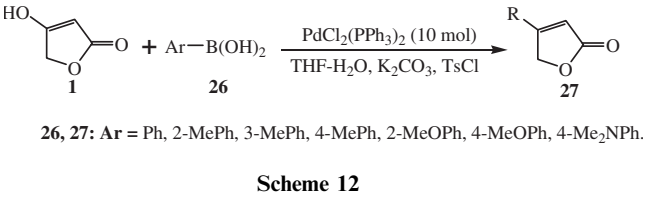
Scheme 9



Scheme 10

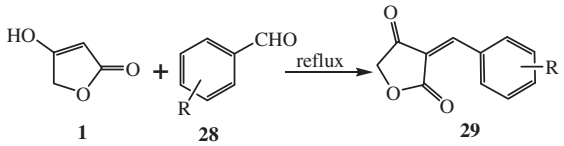
Tetronic acid **1** was dialkenylated with allyl bromide **21** under the phase-transfer catalyst [e.g., benzyltriethylammonium chloride (Kotha and Deb, 2008) or tetra(*n*-butyl) ammonium hydrogensulfate (Kotha et al., 2005)] to give a mixture of C-diallylated tetronic acid **22** and O-allylated isomer **23** [83] (Scheme 10).

4.1.1.3. C_3-benzylation. Jurd (1997, 1996) reported that the reaction of tetronic acid **1** with Mannich bases **24** in acetic acid furnished benzyl lactones **25** (Scheme 11).



26, 27: Ar = Ph, 2-MePh, 3-MePh, 4-MePh, 2-MeOPh, 4-MeOPh, 4-Me_2NPh.

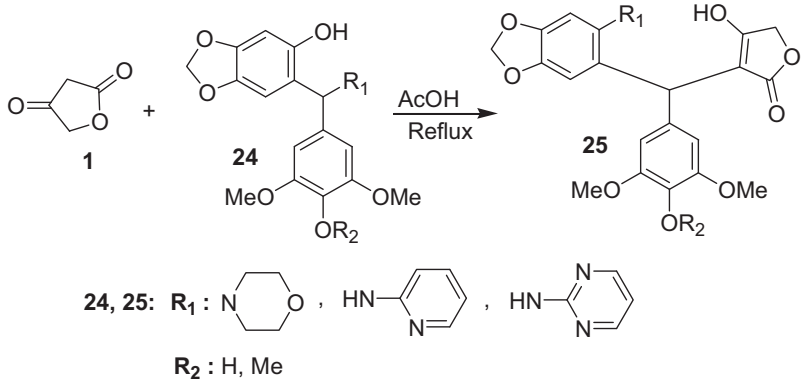
Scheme 12



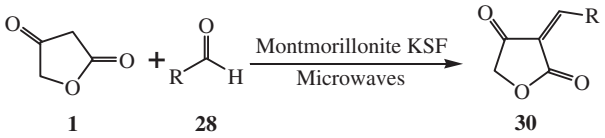
28,29: R = H, Cl, Br, OH, OMe, NMe_2, $N(Ph)_2$, 3-OMe-4-OMe, 3-OEt-4-OMe

Scheme 13

4.1.1.4. Arylation. An efficient palladium-catalyzed cross-coupling reaction of tetronic acid **1** with arylboronic acid **26** in the presence of palladium dichloride in tetrahydrofuran at



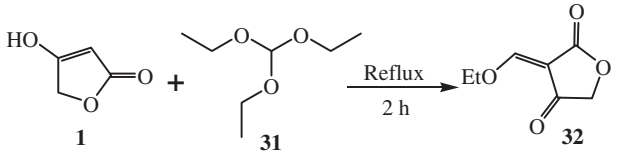
Scheme 11



1 28 30

RCHO: 3,4-methylenedioxybenzaldehyde, 3,4,5-trimethoxybenzaldehyde, 2-thiophenecarboxaldehyde, indole-3-carboxaldehyde, 4-dimetylaminobenzaldehyde, 2-benzyloxybenzaldehyde, 3-(2-furyl)acrolein, ferrocenecarboxaldehyde.

Scheme 14



1 31 32

Scheme 15

60 °C afforded 2(5H)-furanones **27** in good yields (Hu et al., 2011) (Scheme 12).

4.1.1.5. Olefination. A most frequently used method for constructing 3-arylmethylenetetrahydrofuran-2,4-diones **29** is the Knoevenagel condensation between tetronic acid **1** and several substituted benzaldehydes **28** under reflux (Chen et al., 2011; Kozlov et al., 2008; Pashkovskii et al., 2008) (Scheme 13).

The microwave assisted condensation reaction of aldehydes **28** with tetronic acid **1** on acidic Montmorillonite KSF gave efficiently 3-(arylmethylene)-2,4-(3H,5H)furandiones **30** (Villemin and Labiad, 1990) (Scheme 14).

Heating tetronic acid **1** and triethyl orthoformate **31** for two hours gave 3-ethoxymethylene-3*H*-2,4-dione **32** in 60% yield (Otto, 1987) (Scheme 15).

4.1.1.6. Allylindation. Yonemitsu-type trimolecular condensation of **1** with indole **33** and propanal **34** was promoted by $TiCl_4$, afforded 2-(indol-3-yl)-2-(2-propyl)tetronic acid **35** (Gérard et al., 2010) (Scheme 16).

Colombo et al. (2008) developed a three-component domino allylindation reaction of 1*H*-indole-3-carbaldehyde **36** with allyl bromide **37** and **1** in the presence of indium metal in a mixture of tetrahydrofuran and water (1:1) afforded the desired adduct **38** (Scheme 17).

4.1.1.7. Acetylation reaction. C-acylation of tetronic acid **1** with aliphatic carboxylic acids **39** in the presence of *N,N′*-dicyclohexylcarbodiimide (DCC) and 4-dimethylaminopyridine (DMAP) in dichloromethane gave 3-acyltetrahydrofuran-2,4-diones **40** (Baati et al., 2011, 2010; Nomura et al., 1986; Pashkovskii et al., 2003; Rouleau et al., 2013; Murray et al., 2014) (Scheme 18).

4.2. Reactions involving carbon–heteroatom bond formation

4.2.1. C–N bond formation

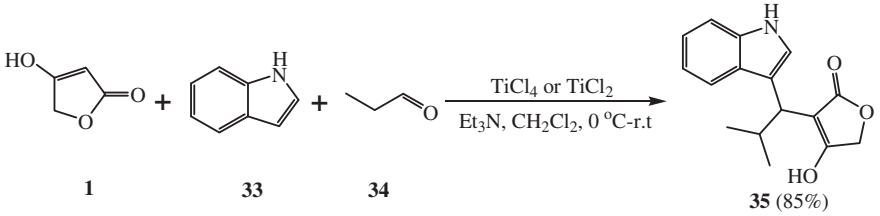
4.2.1.1. Coupling reactions. 3-Arylazotetronic acids **42** were prepared by coupling of **1** with diazotized arylamines **41** in aqueous sodium hydroxide solution (Tanaka et al., 1984) (Scheme 19).

4.2.1.2. Nitration reaction. Nitration of **1** with concentrated nitric acid in diethyl ether afforded 3-nitrotetronic acid **43** (Pollet et al., 1984) (Scheme 20).

4.2.1.3. Amination reaction. Reaction of tetronic acid **1** with an equimolar amount of various anilines **44** produced the corresponding 3-anilinolactones **45** in excellent yields (Cole et al., 2002; Cook et al., 1994; Darwish et al., 2004; Ghahremanzadeh et al., 2013; Jeedimalla et al., 2013; Madec et al., 2008; Momose et al., 1988; Paulvannan and Stille, 1994; Savina et al., 2007; Shaabani et al., 2010) (Scheme 21).

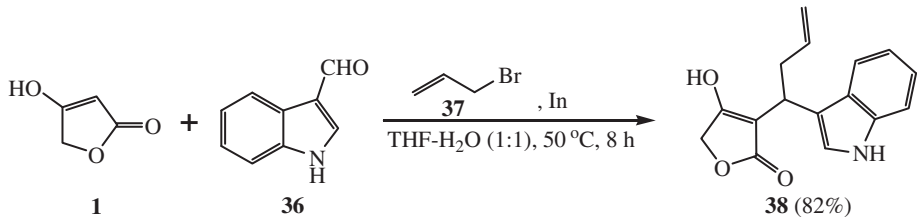
The condensation reaction of tetronic acid **1** with *o*-phenylenediamines **46** at different reaction conditions (YCl_3, AcOH, EtOH) gave 4-(2-aminophenylamino)furan-2(5*H*)-ones **47** (Amari et al., 2002; Bentarzi et al., 2009; Cai et al., 2011; Cheng et al., 2011; Kaoua et al., 2013) (Scheme 22).

4.2.1.4. Diazotization reactions. Regitz reaction using *p*-toluenesulfonyl azide (TsN_3) as the diazo transfer reagent remains the most efficient approach despite it is hampered by potential hazard and purification problems to remove the p-tosylamide co-product (Chapman et al., 1987; Geraghty et al., 2000). Even, 2-azido-3-ethylbenzthiazolium tetrafluoroborate was revealed as an effective diazo transfer reagent for the transformation of **1** into 3-diazotetrahydrofuran-2,4-dione **48** (Stachel et al., 1994) (Scheme 23).

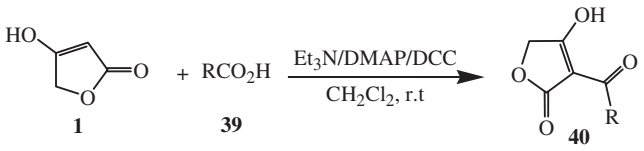


1 33 34 **35** (85%)

Scheme 16

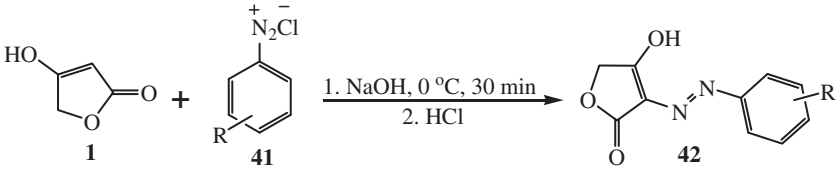


Scheme 17



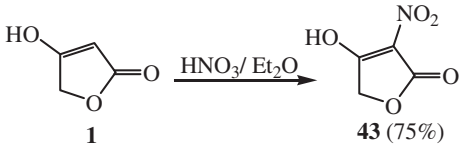
R= $(CH_2)_2Ph$, $CH_2CH{=}CHCH_3$, CH_2OCH_2Ph, $CH_2(CH_2)_2CO_2Me$, $CH_2(CH_2)_2COMe$, $CH_2(CH_2)_4CH_2$, CH_2Br

Scheme 18

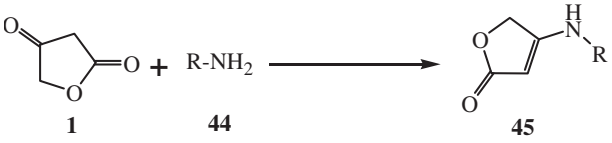


41, 42: R = H, CH_3
X = H, NO2

Scheme 19



Scheme 20



R= Ph, 4-CH_3-Ph, 4-OCH_3-Ph, 4-Br- Ph, 4-Cl- Ph, 4-CH_3- Ph, 4-CO_2H-Ph, 3-OH-Ph, 2-CN-Ph, 2-CO_2H-Ph, 2-CO_2Et-Ph, CH_2Ph

Scheme 21

4.2.2. C–S bond formation

4.2.2.1. Sulfonation reaction. Combs et al. (2005) showed that sulfonation of **1** with sodium hydrogensulfite in ethanol resulted in the formation of the corresponding sulfonic acid **49** (Scheme 24).

4.2.2.2. Thionation reaction. Treatment of **1** with Lawesson's reagent **50** in boiling toluene afforded the regioselective thiolotetronic acid **51** in 70% yield (Desbene-Finck et al., 2006) (Scheme 25).

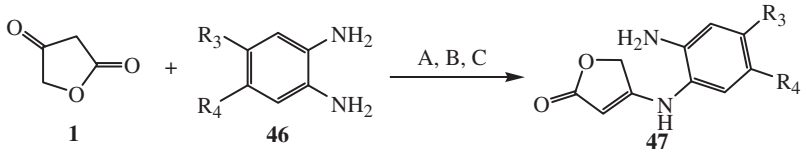
Goddard et al. (2010) have reported that treatment of **1** with Martin's sulfurane **52**, in diethyl ether afforded the corresponding sulfur ylide **53** in 73% yield (Scheme 26).

4.2.3. C–O bond formation

4.2.3.1. Esterification. Radical precursors, enol esters **55**, were prepared by coupling of 2-bromobenzoic acids **54** with tetronic acid **1** in the presence of 2-chloro-1-methylpyridinium iodide and triethylamine in THF at room temperature (Pugh et al., 2003; Zhang, 2000) (Scheme 27).

In addition, O-acylation of tetronic acid **1** with pivaloyl chloride **56** in the presence of 4-dimethylaminopyridine (DMAP) (Axelrod et al., 2013) or 4- pyrrolidinopyridine (Balthazor, 1992) afforded 2,2-dimethyl-2,5-dihydro-5-oxo-3-furanyl ester **57** (Scheme 28).

4.2.3.2. O-alkylation reaction. O-alkyl tetronic acids are compounds of interest, being useful starting materials for natural product synthesis (Hamada and Shiori, 1984; Kametani et al., 1987, 1986; Schmidt et al., 1983). Many methods for the O-alkylation of tetronic acids have been reported in the literature. However, most of the reported methods are restricted to form the methyl and ethyl tetronates using alkylating agents such as dimethyl or diethyl sulfate (Wegnel et al., 1979), trimethyloxonium tetraflouroborate (Wegnel et al., 1979) and diazomethane (Ley and Wadsworth, 1989). The Fischer etherification method (Gelin and Pallet, 1980) was used successfully for the preparation of methyl and ethyl tetronates.



1 **46** **47**

R_3= H, CH_3, Cl, NO_2; **R_4** = H, CH_3, Cl, NO_2

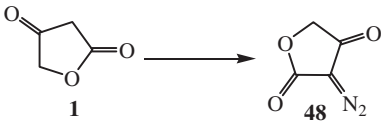
Conditions

A: Ytterbium trichloride in ethanol, Time = 24h, T= 20 °C.

B: Acetic acid in water, Time = 0.25h.

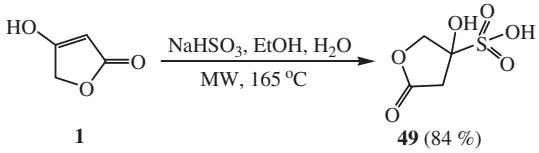
C: Ethanol, Time = 0.5h, Heating.

Scheme 22



1 **48**

Scheme 23



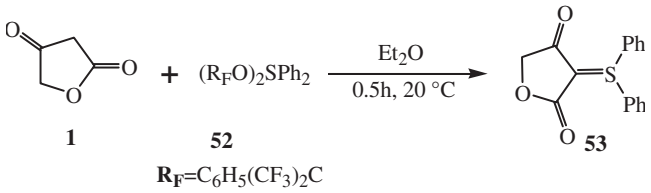
1 **49** (84 %)

Scheme 24

Zimmer et al. (1988) studied the preparation of alkyl tetronates by treatment of primary or secondary alcohols and tetronic acid with conc. H_2SO_4. Although this procedure allows for preparation of various alkyl ethers, it has some serious limitations.

Hoffmann et al. (1989) have reported a modification of Zimmer's method which replaces the excess of conc. H_2SO_4 with a catalytic amount of *p*-toluenesulfonic acid. While this modification works well with primary alcohols, secondary alcohols gave poor yields of the alkyl tetronate.

Treatment of tetronic acid **1** with an alkyl bromides or with alcohols **58** under Mitsunobu conditions [diisopropyl diazenedicarboxylate (DIAD)] (Bach and Kemmler, 2003; Bajwa and Anderson, 1990; Billaud et al., 2007) represents a general method for the regio-specific O-alkylation of tetronic acid **59** in high yields. Alternatively, cesium fluoride (CsF) in dimethylformamide (DMF) has been reported to be an efficient agent for the O-alkylation of tetronic acid (method B) (Bach et al., 2004) (Scheme 29).



1 **52** **53**

R_F=$C_6H_5(CF_3)_2C$

Scheme 26

4.2.3.3. Sulfonates ether formation. Several groups have developed general methodology for the one step formation of **61** *via* tosylation reaction of tetronic acid **1** with tosyl chloride **60** in the presence of triethylamine at room temperature (Blanc et al., 2013; Sun et al., 2005; Wang et al., 2003) (Scheme 30).

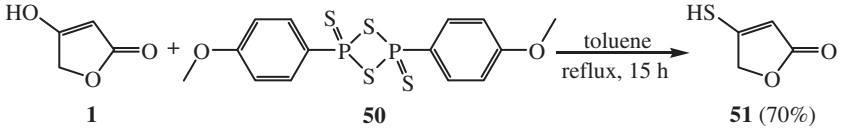
The compound **1** was readily converted to the corresponding triflates **63** in 66% yield by treatment with triflic anhydride **62** in methylene chloride in the presence of *N,N*-diisopropylethylamine (DIPEA) (Grigg et al., 1997, 1994) (Scheme 31).

4.2.3.4. Phosphorylation reaction. 4-Phosphonium trifluoromethanesulfonate **65** was generated *in situ* by the reaction of tetronic acid **1** with triphenylphosphonium anhydride trifluoromethanesulfonate (Hendrickson's reagent) **64** (Paintner et al., 2003) (Scheme 32).

4.2.4. Carbon–halogen bond formation

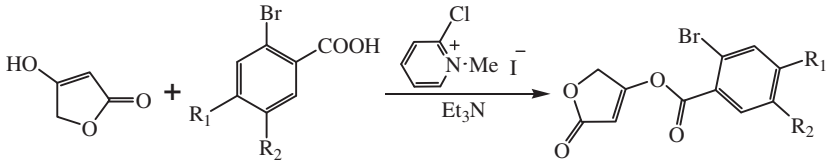
Halogenoheteroarenes are useful intermediates for the syntheses of bioactive natural products and pharmaceutical drugs.

4.2.4.1. Bromination. Bromination of tetronic acid **1** with oxalyl bromide in dichloromethane and DMF at room temperature yielded 4-bromo-5*H*-2-furanone **66** (Boukouvalas and Albert, 2011; Bach et al., 2012; Barrett



1 **50** **51** (70%)

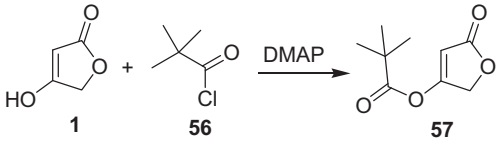
Scheme 25



1 **54** **55**

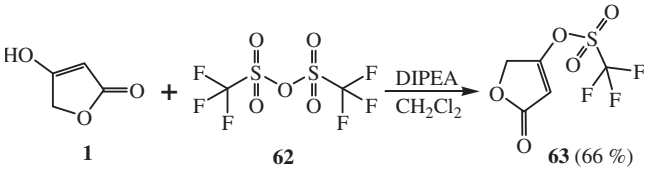
54, 55: $\mathbf{R_1}$ = H, OMe, CH_3, C_4H_9
$\mathbf{R_2}$ = H, OMe.

Scheme 27



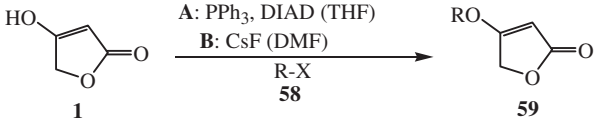
1 **56** **57**

Scheme 28

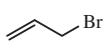
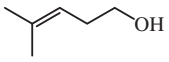
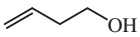
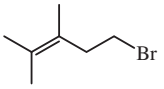
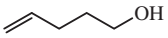
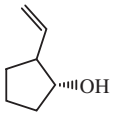
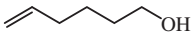
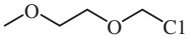
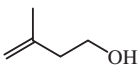


1 **62** **63** (66 %)

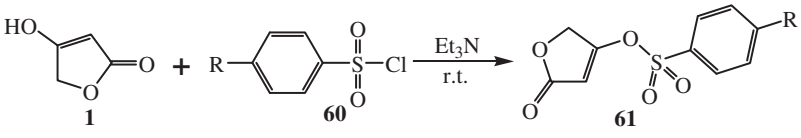
Scheme 31



1 **59**

Comp.	Method	R-X	Yield %	Comp.	Method	R-X	Yield %
58, 59a	B		64	**f**	A		80
b	A		80	**g**	B		86
c	A		85	**h**	A		77
d	A		83	**i**	B		86
e	A		85				

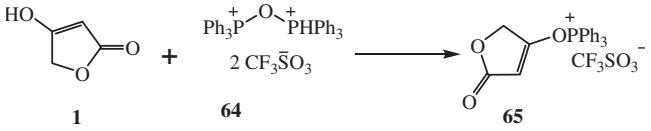
Scheme 29



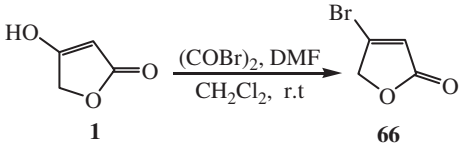
1 **60** **61**

60, 61: R = CH_3, NO_2

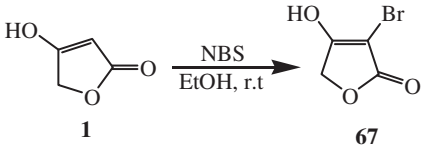
Scheme 30



Scheme 32



Scheme 33



Scheme 34

et al., 2006; Brecker et al., 1999; Jas, 1991; Xin et al., 2000) (Scheme 33).

When the bromination of **1** is carried out using N-bromosuccinimide (NBS) as effective promoters in ethanol at room temperature, it afforded 2-bromotetronic acid 184 (Ge and Kirk, 1997) (Scheme 34).

5. Conclusion

We hope to have conveyed to the readers of this review the current interest of the synthetic community in the synthesis, and chemical reactivity of tetronic acid. It seems likely that this review will serve as a useful reference for chemists interested in discovering new types of reactions and synthesis of tetronic acid.

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