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Dedicated to Professor Dr. Miklós Zsuga on the occasion of his 60th birthday.

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1. Introduction.

The first natural isoflavone, the irigenin glucoside was isolated by Laire and Tiemann in 1893 from the *Iris florentina* [1]. Later, in 1910, the prunetin was defined as an isoflavone type compound [2]. The first synthesis of the isoflavones was initiated by Baker and Robinson in 1925 [3]. The isoflavone name has also been coined by Baker and Robinson. Since the isolation of the first naturally occurring isoflavone derivatives, these oxygen heterocyclic compounds became well known products of the plant kingdom.

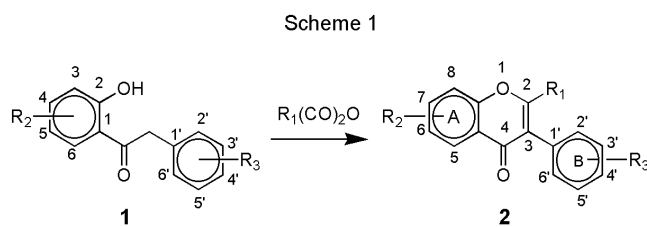
Isolation, structure elucidation and synthesis of the isoflavones have been reviewed both in review articles [4-6] and book chapters [7-10]. Usually the most popular selected synthetic procedures are discussed with the help of limited number of examples. In their account on the synthesis of flavonoids, Wagner and Farkas [8] described several well known methods for the preparation of variously substituted isoflavones.

For this reason, the major aim of this review is to compile most of the synthetic methods used for the preparation of natural, semisynthetic and synthetic isoflavones (3-phenylchromones) in one account. Frequently used and most important procedures are discussed in chronological order of their invention. Because of the huge amount of the published data on this topic, only selected examples are provided to illustrate a special synthetic method *via* several literature references.

2. Synthesis of 2-Substituted Isoflavones.

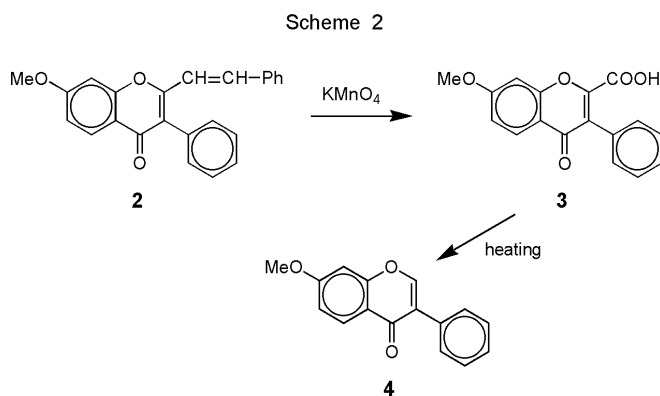
In their seminal paper, Baker and Robinson [3] described the first synthesis of isoflavones by the reaction of 2-hydroxydeoxybenzoins **1** with a mixture of acetic anhydride and sodium acetate, benzoic anhydride and sodium benzoate, and cinnamic anhydride and sodium cinnamate, respectively, to afford 2-substituted isoflavones **2** (Scheme 1). In the case where the isoflavones are synthesized by the ring closure of 2-hydroxydeoxybenzoins, the C-2 atom of the isoflavone molecule should be incorpo-

rated in the course of the ring closure. This method solves this problem by the acylation of the methylene group of the starting 2-hydroxydeoxybenzoins.



R_1 : Me, Ph, Ph-CH=CH-

Since there are no 2-substituted derivatives among the naturally occurring isoflavones, this simple and convenient procedure cannot be used for the preparation of natural isoflavones. However, further chemical transformations of the 2-styrylisoflavones (**2**, R^1 : Ph-CH=CH-) made possible the preparation of 2-unsubstituted isoflavones. 2-Styrylisoflavone **2** was oxidized with potassium permanganate to obtain 2-carboxyisoflavones **3** which gave then 7-methoxyisoflavone **4** on thermal decarboxylation (Scheme 2). Several other 2-unsubstituted isoflavones were synthesized *via* 2-styrylisoflavones [11-18]. Later, more convenient procedures superseded this multistep procedure.

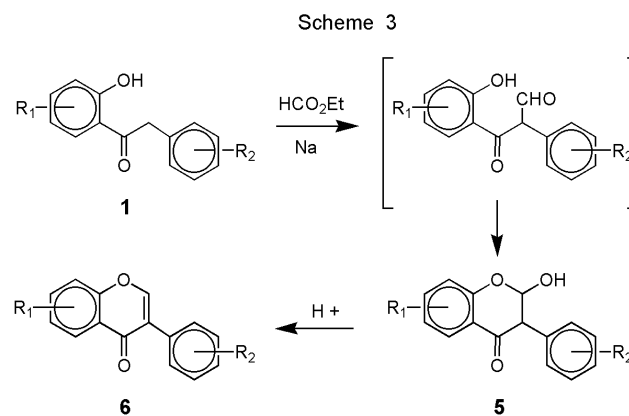


Although the procedure invented by Baker and Robinson for the preparation of 2-substituted isoflavones cannot be used for direct synthesis of natural isoflavones, it remained popular until now to prepare synthetic isoflavone derivatives *e.g.* for drug research. This method has been generalized for the synthesis of variously substituted isoflavones at position 2 [19-36]. This ring closure reaction could also be utilized for the preparation of 2-methylisoflavone glucosides by the reaction of 2-hydroxydeoxybenzoin

glucosides with a mixture of acetic anhydride and sodium acetate [37,38]. To close this chapter, it can be concluded that this simple and versatile procedure remained a popular synthetic method in the isoflavone chemistry for about eight decades.

3. Synthesis of Isoflavones by the Ring Closure of 2-Hydroxydeoxybenzoins with Ethyl Formate and Sodium.

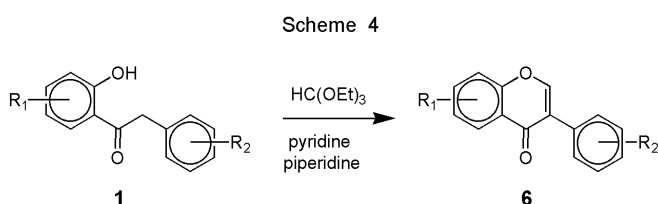
For the preparation of naturally occurring isoflavones such synthetic procedures are convenient which provide 2-unsubstituted isoflavones directly. This requirement can be fulfilled *via* the formylation of the methylene group of 2-hydroxydeoxybenzoins **1**, followed by a ring closure reaction to obtain 2-hydroxyisoflavanones **5** which give then the desired isoflavones **6** on dehydration under acidic conditions (Scheme 3). There are several opportunities for the formylation of the methylene group of the 2-hydroxydeoxybenzoins. Various isoflavone syntheses have been invented by the utilization of this principle.



First synthetic method of this type was worked out by Wessely *et al.* in 1933 [39]. The reaction of the appropriate 2-hydroxydeoxybenzoin **1** in a mixture of anhydrous ethyl formate and powdered sodium provided a well known natural isoflavone, the daidzein (7,4'-dihydroxyisoflavone (**6**, R^1 : 7-OH, R^2 : 4'-OH)). The next examples originate from Venkataraman *et al.* [40] who synthesized several substituted isoflavones by the same method. Later, this simple procedure became a generally used one and numerous natural, semisynthetic and synthetic isoflavones were prepared by using this formylation method [41-60]. The original reaction conditions were more or less modified to improve the utility of this versatile procedure. It is worth mentioning that Indian chemists, first of all the laboratories of K. Venkataraman and T. R. Seshadri, were especially active in the application of this procedure for the synthesis of a wide variety of natural isoflavones.

4. Synthesis of Isoflavones by the Reaction of 2-Hydroxydeoxybenzoins with a Mixture of Ethyl Orthoformate, Pyridine and Piperidine.

Another procedure for the preparation of 2-unsubstituted isoflavones is also based on the formylation of the methylene group of the 2-hydroxydeoxybenzoins **1**. Sathe and Venkataraman invented the utilization of the ethyl orthoformate for this purpose [61]. 2-Hydroxydeoxybenzoins **1** are reacted with a mixture of anhydrous ethyl orthoformate, pyridine and piperidine to afford isoflavones **6** (Scheme 4) *via* 2-hydroxyisoflavanones **5** (Scheme 3) similarly to the use of a mixture of ethyl formate and sodium [39-60].



Later, this procedure has been used by numerous research groups all over the world, mainly for the synthesis of natural isoflavones [62-72]. As a result, it has also turned out that this method is almost insensitive to the substitution pattern of the starting 2-hydroxydeoxybenzoins. This feature renders this Venkataraman's procedure especially beneficial for the synthesis of natural isoflavones with hydroxy and/or methoxy substituents. This ring closure reaction could also be used for the preparation of isoflavone glycosides from 2-hydroxydeoxybenzoin glycosides [37,38,73-75].

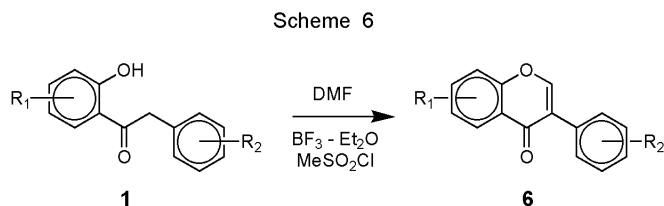
5. Synthesis of Isoflavones with Zinc Cyanide Reagent.

The next synthetic procedure based on the formylation of the methylene group of the 2-hydroxydeoxybenzoins **1** was invented by Farkas in 1957 [76]. In this case zinc

cyanide was used as reagent for the formylation reaction. The appropriate 2-hydroxydeoxybenzoin **1** was allowed to react with zinc cyanide in anhydrous ether with dry hydrogen chloride to afford an imine. This imine was isolated and then hydrolyzed with dilute hydrochloric acid to obtain the desired isoflavone **6** *via* the formyl derivative as described for the above-mentioned related procedures (Scheme 5) [76-78]. However, it should be mentioned that this method has not hitherto received a general acceptance.

6. Synthesis of Isoflavones *via* the Formylation of 2-Hydroxydeoxybenzoins with *N,N*-Dimethylformamide.

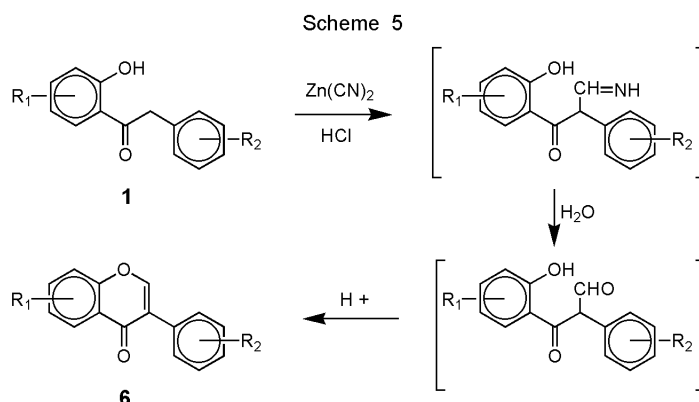
An isoflavone synthesis first published by Bass in 1976 [79] is also based on the formylation of the methylene group of 2-hydroxydeoxybenzoins **1**. The formylating agent is *N,N*-dimethylformamide in this case and then the reaction proceeds as described for the related methods to yield isoflavones **6** (Scheme 6).



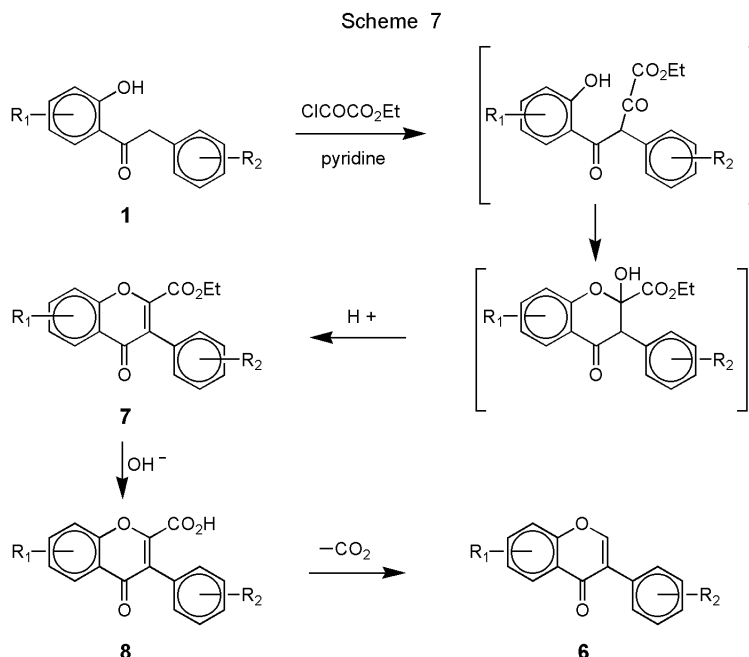
Contrary to the fact that this procedure seems to be a simple one, it has not hitherto received a general acceptance although it has been used even in recent years for the synthesis of a few polyhydroxyisoflavones [80-83]. In special cases this method makes possible the one-pot synthesis of isoflavones starting from the appropriate phenol and phenylacetic acid derivative [80,82].

7. Synthesis and Decarboxylation of 2-Carboxyisoflavones.

A really new synthetic method was published by Baker *et al.* in 1952 [84,85]. This procedure is based on the acylation



of the methylene group of the 2-hydroxydeoxybenzoins similarly to the first isoflavone synthesis invented by Baker and Robinson in 1925 [3]. Experimental details of this method were communicated by Baker *et al.* in their full papers in 1953 [86,87]. 2-Hydroxydeoxybenzoins **1** are allowed to react with ethoxalyl chloride in pyridine at room temperature to give 2-carbethoxyisoflavones **7**. Baker *et al.* suggested a mechanism for this ring closure reaction [86], which is illustrated by Scheme 7.

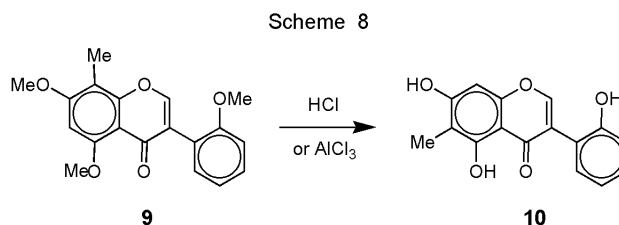


In the case of polyhydroxydeoxybenzoins ethoxalylation of all but the *ortho* hydroxyl groups may take place. However, this fact does not result in any difficulty since these ethoxalyl groups are removed on the acidification of the reaction mixture with a dilute acid to provide 2-carbethoxyisoflavones **7** with free hydroxyl groups. Compounds **7** can be saponified by mild alkaline hydrolysis to obtain 2-carboxyisoflavones **8**. These 2-carboxy derivatives are then converted into the appropriate isoflavones **6** by decarboxylation at their melting points. The general applicability of this method is illustrated by the fact that numerous naturally occurring isoflavones were synthesized using this procedure. This method became popular almost immediately and was successfully used by numerous research groups [88-101] for the synthesis of variously substituted isoflavones **6**. In some cases the 2-carbethoxyisoflavones have also been utilized in the drug research [99,100].

8. Preparation of Isoflavones by Ring Isomerization.

Although several procedures were known for the synthesis of isoflavones in the middle of the twentieth century, there were difficulties concerning the synthesis of isoflavones of certain substitution pattern of their ring A. Such a difficulty can be overcome by ring isomerization. On the ring isomerization of the isoflavones the first papers were published in 1953 and 1954 [52,102-104]. Demethylation of the 5,7,2'-trimethoxy-8-methylisoflavone (**9**) with hydrochloric acid or aluminium chloride

furnished 5,7,2'-trihydroxy-6-methylisoflavone (**10**) (Scheme 8) as a result of demethylation and ring isomerization [52,102]. Similar ring isomerization was observed by using hydrobromic acid [103] or hydroiodic acid [104] for demethylation of the methoxy groups of methoxyisoflavones.



In 1959, Dhar and Seshadri published the synthesis of 5,6,7-trisubstituted isoflavones by potassium hydroxide mediated ring isomerization of 5,7,8-trisubstituted

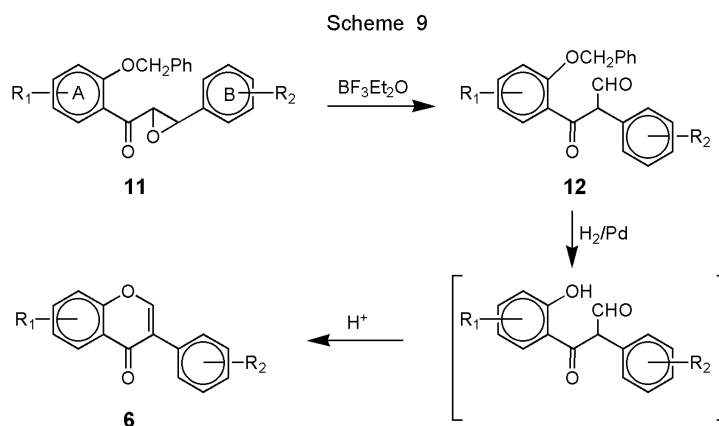
isoflavones [105]. Potassium ethoxide induced ring isomerization of the isoflavones was thoroughly studied in the laboratory of Farkas and the syntheses of numerous natural isoflavones were successfully accomplished by this research group with the help of this chemical transformation of variously substituted isoflavones [58,106-118]. In some cases even 5,7,8-trisubstituted isoflavone glycosides could be isomerized into 5,6,7-trisubstituted isoflavone glycosides with potassium carbonate in alcoholic solution [119,120].

9. Synthesis of Isoflavones by the Conversion of 2'-Benzyloxychalcone Epoxides.

A common feature of the above-discussed isoflavone syntheses is that the starting material is a 2-hydroxydeoxybenzoin in each case and the C-2 carbon atom of the isoflavone skeleton should be inserted in the course of the ring closure reaction. This insertion can be accomplished by the formylation or acylation of the methylene group of

the 2-hydroxydeoxybenzoin as a first step, ring closure of which provides an isoflavone as in the case of all the procedures based on the formylation of the methylene group of the 2-hydroxydeoxybenzoin. For the conversion of chalcones into isoflavones, the first experiments were performed in the laboratory of Seshadri about forty years ago [121-125]. 2'-Benzyloxychalcone epoxides **11** were rearranged into α -formyldeoxybenzoin with boron trifluoride etherate. Compounds **12** were then debenzylated either by catalytic hydrogenation or by treatment with hydrochloric acid-acetic acid mixture to afford isoflavones **6** (Scheme 9). Seshadri *et al.* managed to synthesize numerous naturally occurring isoflavones in this way.

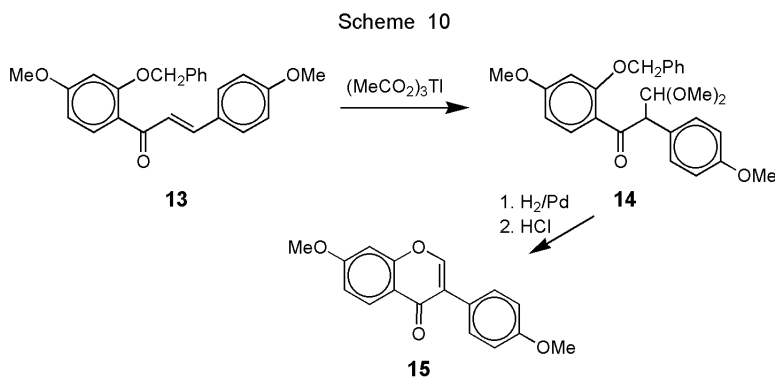
This rearrangement of the 2'-benzyloxychalcone epoxides was successfully utilized by others to prepare variously substituted isoflavones [126]. Litkei and Bognár [127,128] studied the correlation between the Hammett σ



the 2-hydroxydeoxybenzoin with various reagents as described in the preceding chapters.

In case the starting materials are chalcone derivatives, all the carbon atoms of the isoflavone skeleton are present in the starting material. The appropriate chalcone deriva-

values of the substituents in the ring B of the starting 2'-benzyloxychalcone epoxides and the types of the flavonoid derivatives obtained on the conversion of the epoxides. As a result, it was concluded that the presence of an electron donor substituent in the ring B favours the



isoflavone formation in the course of the rearrangement of the 2'-benzyloxychalcone epoxides.

10. Synthesis of Isoflavones by Thallium(III) Nitrate Oxidation of 2'-Hydroxychalcones.

Another opportunity to convert chalcones into isoflavones is the oxidation of 2'-hydroxychalcones with thallium salts. First example of this conversion was published by Ollis *et al.* in 1968 [129]. The thallic acetate oxidation of 2'-benzyloxy-4,4'-dimethoxychalcone (**13**) in methanol yielded the acetal **14**. Hydrogenolysis and subsequent hydrolysis with ethanolic hydrochloric acid combined with ring closure provided 7,4'-dimethoxyisoflavone (**15**) (Scheme 10).

Later, the research group of Ollis performed this oxidation of variously substituted 2'-hydroxychalcones with thallium(III) acetate [130,131]. On the basis of their experimental results, it has been concluded that a 1,2-aryl migration provides the acetal, which is the indispensable intermediate of the isoflavone ring formation. They suggested two mechanisms for the formation of the acetal [130].

In 1970, McKillop *et al.* described the utilization of the thallium(III) nitrate for the oxidative rearrangement of various olefins [132]. This oxidant was successfully used for the similar conversion of 2'-hydroxychalcones into α -formyldeoxybenzoin acetals [133] as performed by Ollis *et al.* [129-131] with thallium(III) acetate. This invention of McKillop *et al.* paved the way for a simple and convenient

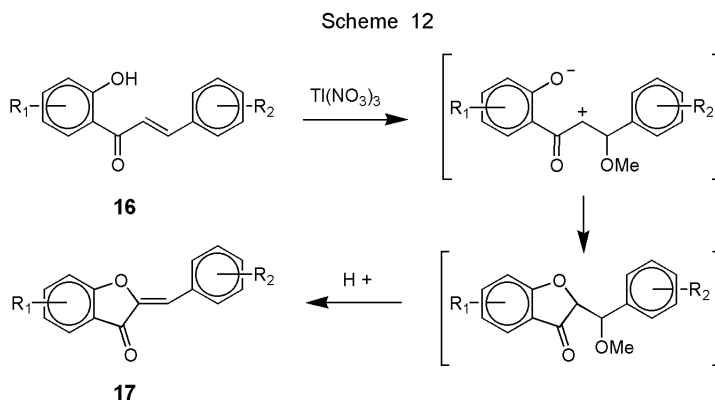
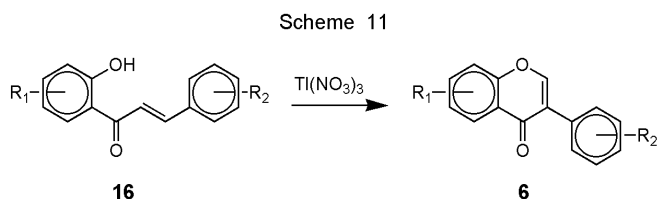
isoflavone synthesis. Later, McKillop discussed the mechanism and utilization of the thallium(III) nitrate oxidation of various olefins with the chalcones among them [134-136].

As far as the utilization of the thallium(III) nitrate oxidation of 2'-hydroxychalcones **16** for the synthesis of naturally occurring isoflavones is concerned, a decisive contribution was provided by the laboratory of Farkas [137-142]. The original reaction conditions were appropriately modified and the procedure was generalized according to the requirements of the preparation of isoflavones with various substitution patterns in their rings A and B (Scheme 11).

Later, this versatile procedure was utilized by several research groups to prepare a wide variety of natural and synthetic isoflavones [143-151]. This method has been successfully used for the synthesis of isoflavones with unsubstituted ring A [152-154] as well. Moreover, *C*-glycosyl- [155] and *O*-glycosylisoflavones [156] were synthesized by the thallium(III) nitrate oxidation of the appropriate 2'-hydroxychalcone *C*-glycosides and *O*-glycosides.

Although the thallium(III) nitrate oxidation of the 2'-hydroxychalcones usually provides isoflavones **6**, in some special cases a mixture of isoflavones **6** and aurones **17** or aurone **17** as sole isolable product are obtained. First example for the aurone formation by the oxidation of 2'-hydroxychalcones **16** with thallium(III) nitrate was described by Lévai and Tókécs in 1982 [157]. A mechanism has also been suggested for this transformation (Scheme 12). Later, this oxidative transformation of the 2'-hydroxychalcones **16** for the synthesis of variously substituted aurones **17** was successfully utilized by other laboratories [158-161].

To close this chapter, it can be concluded that the thallium(III) nitrate oxidation of the 2'-hydroxychalcones **16** is probably the most versatile procedure for the synthesis of variously substituted isoflavones. One of its benefits is that the starting 2'-hydroxychalcones are usually easily available compounds.



11. Preparation of Isoflavones by Conversion of Flavanones into Isoflavones.

First examples for the conversion of flavanones **18** into isoflavones **6** was reported about four decades ago [162,163]. This method belongs to those types of procedures when all the carbon atoms of the isoflavone skeleton are present in the starting material and an appropriate rearrangement provides the isoflavone structure. Several possible routes were supposed by Pelter for the aryl migration taking place in the course of the conversion of flavanones into isoflavones [164].

In 1990, Kinoshita *et al.* [165] performed a laboratory analogy for the *in vivo* conversion of flavanones **18** into isoflavones **6** by thallium(III) nitrate in a mixture of methanol and chloroform. Suggested mechanism of this thallium(III) mediated rearrangement is illustrated by Scheme 13. In the first step, enolization followed by alkoxythallation of the flavanones **18** leads to an unstable intermediate. Dethallation and an aryl migration provide the

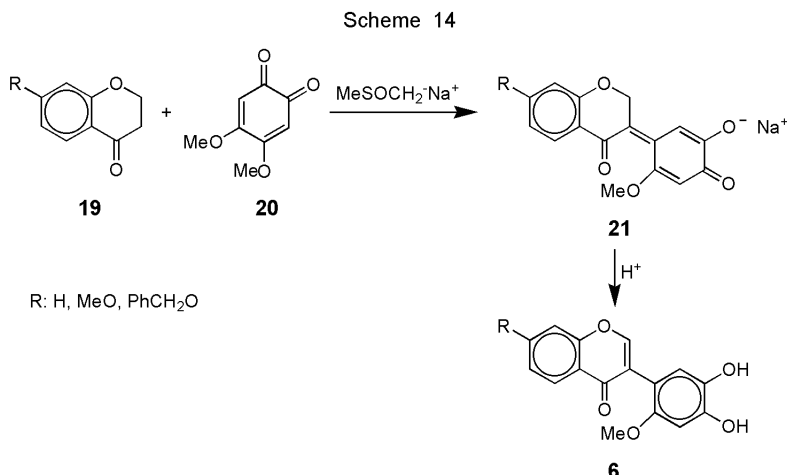
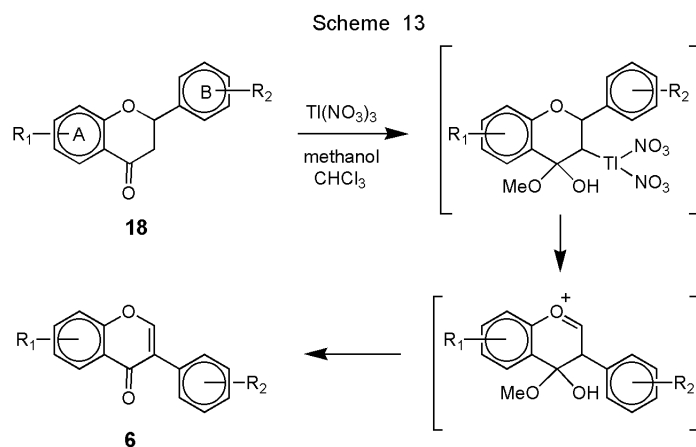
appropriate isoflavones **6** (Scheme 13). Flavanones **18** have also been converted into isoflavones **6** by thallium(III) toluene-*p*-sulfonate in acetonitrile or in propionitrile [166].

Crombie and Whiting described the P 450 enzymic conversion of flavanones into isoflavones [167]. Flavanones have also been rearranged into isoflavones by hypervalent iodine oxidation [168,169].

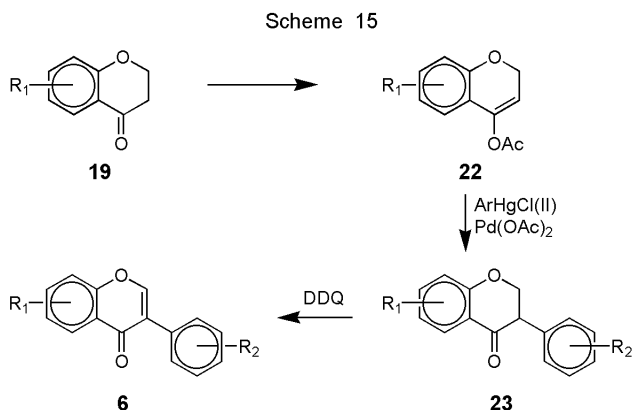
However, this rearrangement has not hitherto become a rational synthetic procedure for the preparation of isoflavones. Nonetheless, it may help the better understanding of the biosynthesis of isoflavones.

12. Preparation of Isoflavones by the Arylation of 4-Chromanones.

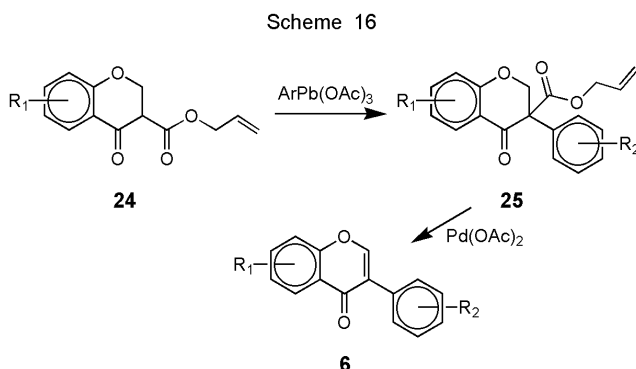
Arylation of the 4-chromanones can also be used for the preparation of isoflavones. Several independent methods can be collected into this group of synthetic procedures. Reaction of 4-chromanones **19** with 4,5-dimethoxy-*o*-benzoquinone (**20**) in anhydrous dimethyl sulphoxide provided quinonmetides **21** which gave then isoflavones **6** on acidification (Scheme 14) [170,171].



Arylation of 4-chromanones **19** may take place *via* their enol acetates **22** affording isoflavanones **23** as isolable products (Scheme 15) [172]. Compounds **23** can then be oxidized with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) to obtain the desired isoflavones **6** (Scheme 15) [173].

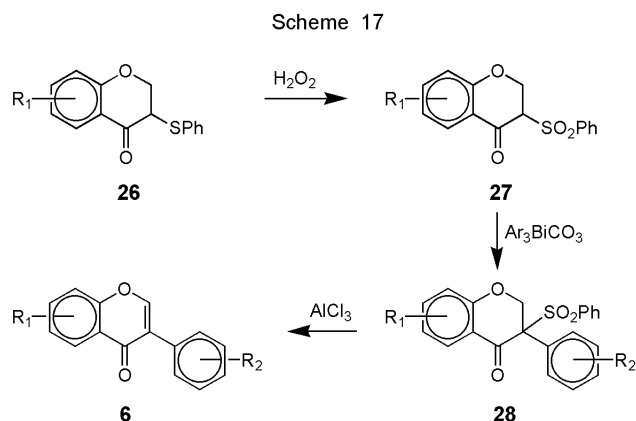


Arylation of 3-(allyloxycarbonyl)-4-chromanones **24** has also been utilized for the preparation of isoflavones **6** [174-177]. An organobismuth-mediated coupling reaction was used for this purpose, but the utility of this method is strongly limited by the difficult availability of the arylbismuth(V) reagent [174]. Aryllead(IV) triacetate proved to be a convenient reagent providing isoflavanones **25** which yielded isoflavones **6** on oxidation with catalytic amounts of palladium(II) acetate and 1,2-bis(diphenylphosphino)ethane (DPPE) in acetonitrile (Scheme 16) [175,176].



Santhosh and Balasubramanian [178,179] have introduced a new procedure for the preparation of 3-alkylchromones and isoflavones starting from 3-phenylthio-4-

chromanones **26** which first were oxidized to the appropriate sulphones to activate the reaction of the C-3 atom. Compounds **27** were then alkylated or arylated to afford 3,3-disubstituted 4-chromanones **28**. Chromanone derivatives **28** gave 3-alkylchromones or isoflavones **6** on treatment with anhydrous aluminium trichloride in methylene chloride (Scheme 17). Donnelly *et al.* modified this method by using aryllead(IV) triacetates as arylating reagents [180].



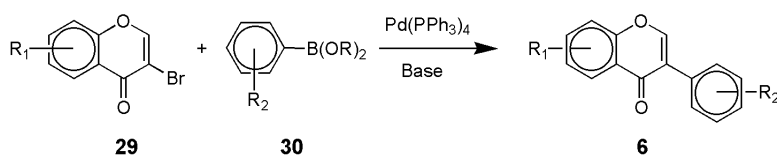
To close this chapter, it is worth mentioning that isoflavones **6** have also been synthesized by the palladium-mediated arylation of 3-bromochromones **29** with arylboronic acids or esters **30** (Scheme 18) [181].

13 Miscellaneous Synthetic Procedures.

In the preceding chapters, synthesis of isoflavones **6** by the formylation of 2-hydroxydeoxybenzoins **1** with generally utilized methods are discussed in detail. However, there are some rarely used formylating procedures that are worth mentioning *via* a few examples.

The modified Vilsmeier-Haack reaction where a mixture of dimethylformamide and phosphorus oxychloride is used as formylating agent has also been utilized to synthesize isoflavones **6** by the ring closure of 2-hydroxydeoxybenzoins **1** [182]. A mixture of ethyl orthoformate and perchloric acid was successfully used for this purpose as well [183]. Krishnamurty and Siva Prasad [184] accomplished the formylation of the 2-hydroxydeoxybenzoins **1** with *N*-formyl imidazole in anhydrous tetrahydrofuran. The 1,3,5-triazine can also be used for the ring closure of 2-hydroxydeoxybenzoins **1** to afford isoflavones **6** [185]. Isoflavones **6** have also been prepared by the reaction of 2-hydroxydeoxybenzoins **1** with acetic-formic anhydride [186]. As a

Scheme 18

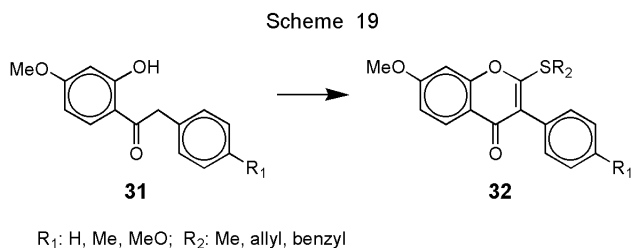


modified version of the original Venkataraman's procedure [61], a mixture of triethyl orthoformate, *N,N*-dimethylformamide and piperidine can also be used for the ring closure of 2-hydroxydeoxybenzoin **1** to provide isoflavones **6** [187].

Oxidation of 2'-hydroxychalcones **16** with phenyliodine(III) bis(trifluoroacetate) (PIFA) has also been utilized for the synthesis of isoflavones **6** [188,189].

Recently, the ring closure of 2-hydroxydeoxybenzoin **1** to obtain isoflavones **6** has been performed under microwave assisted reaction conditions [190,191]. 2-Hydroxydeoxybenzoin **1** have also been converted into isoflavones **6** by using solid phase synthetic method [192].

Kim and Brueggemeier worked out a simple one-pot procedure for the synthesis of 2-(alkylthio)isoflavones **32** by utilizing the phase transfer catalyzed ring closure of 2-hydroxydeoxybenzoin **31** (Scheme 19) [193].



14. Closing Remarks.

In our present review article, synthetic procedures used for the preparation of isoflavones, an important group of naturally occurring oxygen-heterocyclic compounds have been compiled. Our aim was to illustrate each well-known and important method *via* adequate examples described in the selected papers. Over the most popular procedures, several rarely used methods have also been included to try to show the great efforts that have been devoted to this synthetic problem. To the best of our knowledge, this account is the first comprehensive review article on the syntheses of isoflavones published in a chemical journal.

Selected papers have been included as references to help the reader to find original literature data concerning the synthesis of a particular isoflavone.

Acknowledgement.

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