# Benzofuran Systems. Synthesis and Biological Examination of 1-(3-Benzofuranyl)-2-phenylethanones

Halina Kwiecień and E. Baumann\*

Department of Organic Synthesis, Technical University of Szczecin, 71-065 Szczecin, Poland \*Hauptlaboratorium, BASF-AG, 67056 Ludwigshafen, Germany

Received November 22, 1996

Novel 1-(3-benzofuranyl)-2-phenylethanones 4a-d have been prepared by acetylation of 2-alkylbenzofurans 2a-c with phenylacetyl chlorides 3a-b. The methoxy derivatives 4b-d have been demethylated to the corresponding phenols 5b-d with pyridinium hydrochloride. An attempt to obtain the derivatives of 4d and 5a iodinated in the phenyl ring has been undertaken. The novel compounds have been characterized by ir and nmr spectra and their biological activity examined.

J. Heterocyclic Chem., 34, 1587 (1997).

Much attention has been focused on the synthesis of benzofuranyl benzyl ketones because they usually posses interesting pharmacological and other bioactivities [1,2,3]. A number of benzofuranyl phenyl methanones and corresponding carbinols have been synthesized in recent years and tested for various activities, *i.e.* antiarrhythmic [4], antihypertensive [5], antianginal and hypolipenic [6], antibacterial [7] and fungicidal [8]. In this paper the synthesis of novel 1-[3-(2-akylbenzofuranyl)]-2-phenylethanones have been described. These compounds have been considered as key starting materials for the preparation of various new benzofuran derivatives, including products of halogenation, reduction and reductive amination.

The title compounds have been obtained by the Friedel-Crafts acylation of alkylbenzofurans 3a-c with phenylacetyl chlorides 2a-b (Scheme 1). The desired alkylbenzofurans have been prepared by known method using 2-(o-formylphenoxy)alkanoic acids [9,10]. Acids 1a-b, likewise previously obtained 1c [11], were prepared with good yields in the crystalline form by hydrolysis of 2-(o-formylphenoxy)alkanoates [12], and characterized by spectroscopic methods.

The Friedel-Crafts acylation of 2-alkylbenzofurans with phenylacetyl chlorides 2a-b was carried out at 5-10°, using either tin tetrachloride (method A) or aluminium chloride (method B) as a catalyst. The catalyst was added dropwise (tin tetrachloride) or in small portions (aluminium chloride) to the 1,2-dichloroethane solution of the reagents. The reaction carried out by method A took 2 hours to give 57-67% yield of the desired product 4. However, extending the reaction time caused the formation of polymeric products. On the other hand the polymerization process involved in the reaction carried out for 4-5 hours by method B leading to 4 in 60-65% yield appeared to be considerably lower than that observed in method A. It was found that when the reaction mixture was allowed to stand overnight, even at low temperature, the quantity of polymers increased both in the case of method A and B. In spite of the fact that both dosing of liquid tin chloride and decomposition of tin complex with water were convenient, the use of aluminium chloride as the catalyst appeared to be more reliable on account of its low price and low polymerization.

All of the benzofuranyl phenyl ethanones obtained in this work were stable, high-boiling substances and could

OCH<sub>3</sub>

C<sub>4</sub>H<sub>9</sub>

be distilled easily under high vacuum without decomposition. Compound 4b was obtained as pale yellow crystals, the others were light-yellow oils. The structure of the compounds synthesized was determined on the basis of their ir and <sup>1</sup>H nmr spectra.

Methoxyphenyl derivatives 4b-d were demethylated with pyridine hydrochloride to the corresponding phenols 5a-c. The reaction was carried out by refluxing 4b-d with an excess of pyridine hydrochloride either without any solvent or in dry quinoline [13,14]. It was found that in quinoline the reaction was running very slow and the overall yield of product 5 was less than 20%. When the demethylation was carried out without solvent, product 5 was obtained in good yield (60-70%) providing the optimal reaction time was 7-12 minutes (see Experimental). When the reaction time was shorter than optimal phenols 5 obtained were admixed with the starting benzofuran. On the other hand, a longer time was found not to be suitable because of the formation of polymers. Crude demethylation products were light yellow precipitates, which formed colorless crystals with sharp melting points after recrystalization from methanol. The structure of the new compounds 5a-c was confirmed by elemental analysis and characterized by ir and <sup>1</sup>H nmr spectra.

The iodination of 4d and 5a led to products iodinated on their phenyl rings, in spite of the methylene protons being activated by the carbonyl group.

When an ethanol solution of 1-[3-(2-butylbenzofuranyl]-2-(4-methoxyphenyl)ethanone (4d) was treated at 60° with iodine in ethanol in the presence of catalytic amounts of sulphuric acid and with the use of perhydrol as an oxidizing agent, the mono iodo derivative 6a was obtained in 50% yield. Under similar conditions the corresponding hydroxyethanone 5d gave the diiodo compound 7a in 34% yield. On the other hand, the reaction of an alkanoic solution of 5d with iodine in a potassium hydroxide solution gave a mixture of iodinated compounds in the form of an amorphous, alkali-insoluble precipitate. Their ir and <sup>1</sup>H nmr spectra showed that the product mixture contains no hydroxyl group but diiodinated ethers of 5d. The benzofuranyl methoxy- and hydroxyphenylethanones obtained have been screened for their pharmaceutical, fungicidal, herbicidal, and insecticidal activities. None of the compounds examined showed good pharmaceutical or plant protection activity. Ethyl methoxy 4b and ethyl hydroxy 5a showed only weak activity on lemna in laboratory tests.

## **EXPERIMENTAL**

Melting points were determined on a Boetius apparatus and were not corrected. The ir spectra were recorded on a Specord M

80 Carl Zeiss, Jena spectrophotometer. The  $^{1}$ H nmr spectra were recorded on a TM Bruker DPX 400 spectrometer, in deuterio-chloroform as a solvent. Chemical shifts were reported as  $\delta$  values (ppm) downfield from internal tetramethylsilane. Pyridine hydrochloride was purchased from Merck Chemical Co. Commercial 1,2-dichloroethane was distilled from phosphorus pentoxide before use. Phenylacetyl chloride and p-methoxy-phenylacetyl chloride were obtained by a standard procedure from the corresponding acids and thionyl chloride as oils, bp  $[^{\circ}$ C/mm Hg]:132/12; 150-152/14, 3b, bp 143/10 lit [15]. Methyl 2-(o-formylphenoxy)butanoate, pentanoate and hexanoate were prepared as previously described [11,12].

General Procedure for Synthesis 2-(2-Formylphenoxy)alkanoic Acids 1a-c.

A mixture of methyl 2-(o-formylphenoxy)alkanoates (0.5 mole), potassium hydroxide (39.2 g, 0.7 mole), 350 ml of water and 30 ml of methanol was stirred and heated on a steam bath for 2 hours. The solution was cooled, acidified with 10% hydrochloric acid, and the resulting oil or precipitate was separated. The crude product was dissolved in 5% sodium bicarbonate and then was mixed with activated carbon. The mixture was filtered and next acidified to give crystalline acids.

## 2-(2-Formylphenoxy)butanoic Acid (1a).

This compound was obtained as colorless needles (methyl alcohol), 93.7 g (90%), mp 98-99°; ir (potassium bromide):  $\nu$  1640, 1650 (C=O), 1730 (C=O), 2500-3200 (OH) cm<sup>-1</sup>;  $^{1}$ H nmr:  $\delta$  1.13 (t, 3H, CH<sub>3</sub>, J = 7.4 Hz), 2.12 (m, 2H, CH<sub>2</sub>), 4.78 (t, 1H, CH, J = 5.7), 6.90 (d, 1H arom, J = 7.5), 7.09 (t, 1H arom, J = 7.5), 7.53 (t, 1H arom, J = 7.0, 1.6), 7.84 (dd, 1H, arom, J = 7.0, 1.7), 10.21 (br s, 1H, COOH), 10.47 (s, 1H, CHO).

Anal. Calcd. for  $C_{11}H_{12}O_4$  (208.21): C, 63.45; H, 5.81. Found: C, 63.40; H, 5.97.

## 2-(2-Formylphenoxy)pentanoic Acid (1b).

This compound was obtained as pale yellow needles, 96.7 g (87%), mp 79-80°; ir (potassium bromide): v 1650, 1660 (C=O), 1730 (C=O), 2500-3200 (OH) cm<sup>-1</sup>,  $^{1}$ H nmr (deuteriochloroform):  $\delta$  1.00 (t, J = 7.3 Hz, 3H, CH<sub>3</sub>), 1.58 (m, 2H, CH<sub>2</sub>), 2.08 (m, 2H, CH<sub>2</sub>), 4.83 (t, J = 4.9, 1H, CH), 6.90 (d, J = 8.4, 1 H arom), 7.10 (t, 1 H arom, J = 7.5), 7.53 (t, 1H, arom, J = 7.0, 1.7) 7.84 (dd, J = 7.0, 1.7, 1H arom) 9.49 (br s, COOH), 10.47 (s, CHO).

Anal. Calcd. for  $C_{12}H_{14}O_4$  (222.24): C, 64.85; H, 6.35. Found: C, 64. 67; H, 6.55.

## 2-(2-Formylphenoxy)hexanoic Acid (1c).

This compound was obtained as colorless crystals, mp 76-78°; ir and <sup>1</sup>H nmr reported [11].

General Procedure for the Synthesis 2-Alkylbenzofurans 2.

2-Ethyl and 2-propylbenzofurans were obtained by using the procedure previously described for the preparation of 2-butylbenzofuran [11].

#### 2-Ethylbenzofuran 2a.

This compound was obtained as a colorless liquid, 62%, bp 69°/5 mm Hg, lit [9] bp 102°/15 mm Hg;  $^{1}$ H nmr:  $\delta$  1.20 (t, 3H, CH<sub>3</sub>, J = 7.5), 2.55 (q, 2H, CH<sub>2</sub>, J = 7.4), 6.26 (s, 1H, furanyl), 7.01-7.50 (m, 4H, phenyl protons).

Anal. Calcd. for  $C_{10}H_{10}O$  (146.19): C, 82.16; H, 6.89. Found: C, 82.35; H, 6.98.

## 2-Propylbenzofuran 2b.

This compound was obtained as colorless liquid, 58%, bp 75-76°/5 mm Hg, lit [9] bp  $108-115^{\circ}/15$  mm Hg;  $^{1}$ H nmr:  $\delta$  1.00 (t, 3H, CH<sub>3</sub>, J = 7.4), 1.76 (sextet, 2H, CH<sub>2</sub>, J = 7.4), 2.73 (t, 2H, CH<sub>2</sub>, J = 7.4), 6.36 (s, 1H, furanyl), 7.19 (m, 2H, phenyl), 7.39 (d, 1H, phenyl, J = 7.5), 7.46 (d, 1H, phenyl, J = 6.7, 1.7).

*Anal.* Calcd. for  $C_{11}H_{12}O$  (160.22): C, 82.46; H, 7.55. Found: C, 82.22; H, 7.70.

General Procedure for the Reaction of 2-Alkylbenzofurans 2a-c with Phenylacetyl Chlorides 3a-b.

#### Method A.

To a stirred and cooled (5-10°) solution of 2-alkylbenzofuran (2) (0.1 mole) and phenacetyl chloride 3a (0.1 mole) in dry 1,2-dichloroethane (100 ml), anhydrous tin tetrachloride (24 ml) was added dropwise for forty minutes. After the reaction mixture was stirred for an additional two hours at 5-10°, and cooled to 0°, water (200 ml) was added dropwise. The mixture was stirred for forty minutes. The 1,2-dichloroethane organic phase was separated, washed with water, dried over magnesium sulfate and evaporated to give product 3 as an oil, which was purified either by vacuum distillation or recrystallization from methanol.

#### Method B.

To a stirred and cooled (5-10°) solution of 2-alkylbenzofuran (1) (0.05 mole) and phenylacetic chloride (2) (0.05 moles) in dry 1,2-dichloroethane (100 ml) anhydrous aluminium chloride (6.6 g, 0.05 mole) was added portionwise for 1 hour and then the reaction mixture was stirred at that temperature for four hours. The mixture was then poured into water (20 ml) with ice (20 g) and hydrochloric acid (7 g). The organic layer was separated, dried and the solvent removed *in vacuo* to leave a brown oil, which was purified by method A.

## 1-[3-(2-Butylobenzofuranyl)]-2-phenylethanone (4a).

This compound was obtained as a pale yellow oil, yield, method A 65%; method B 60%, bp 174-176°/1 mmHg; ir (carbon tetrachloride): v 1680 (C=O) cm<sup>-1</sup>; uv: <sup>1</sup>H nmr: δ 0.94 (t, 3H, CH<sub>3</sub>, J = 7.3), 1.40 (m, 2H, CH<sub>2</sub>), 1.74 (m, 2H, CH<sub>2</sub>), 3.15 (t, 2H, CH<sub>2</sub>, J = 7.6), 4.29 (s, 2H, CH<sub>2</sub>CO), 7.23-7.36 (m, 7H, arom), 7.47 (m, 1H, arom), 7.96 (m, 1H, arom).

Anal. Calcd. for  $C_{20}H_{20}O_2$ : C, 82.16; H, 6.89. Found: C, 82.04; H, 6.97.

## 1-[3-(2-Ethylbenzofuranyl)]-2-(4-methoxyphenyl)ethanone (4b).

This compound was obtained from 2a and 3b according to Method A as a dark-yellow oil which was purified by distillation in vacuo bp 185-186/1 mm Hg, next crystallized from hexane giving colorleess prisms (57%).

#### Method B.

Yellow crystals (65%), were obtained which were crystallized from methanol, mp 55-56° ir (carbon tetrachloride): v CO 1670 cm<sup>-1</sup>;  $^{1}$ H nmr:  $\delta$  1.34 (t, J = 7.5 Hz, 3H, CH<sub>3</sub>), 3.18 (q, J = 7.5 Hz, 2H, CH<sub>2</sub>), 3.79 (s, 3H, OCH<sub>3</sub>), 4.23 (s, 2H, CH<sub>2</sub>), 6.88 (dd, J = 8.4, 2H, phenyl), 7.17 (dd, J = 8.4, 2H, phenyl) 7.33 (m, 2H, benzofuranyl), 7.46 and 7.96 ppm (2m, 2H, benzofuranyl).

Anal. Calcd. for  $C_{19}H_{18}O_3$  (294.35): C, 77.53; H, 6.16. Found: C, 77.38; H, 6.32.

1-[3-(2-Propylbenzofuranyl)]-2-(4-methoxyphenyl)ethanone (4c).

This compound was obtained from **2b** and **3b** by Method A in 62% yield and by Method B in 60% yield as a yellow oil, bp 185-188°/1 mm Hg; ir (carbon tetrachloride): v CO 1680 cm<sup>-1</sup>;  $^{1}$ H nmr:  $\delta$  1.01 (t, 3H, CH<sub>3</sub>, J = 7.4), 1.79 (sextet, 2H, CH<sub>2</sub>, J = 7.4), 3.13 (t, 2H, CH<sub>2</sub>, J = 7.6), 3.78 (s, 3H, OCH<sub>3</sub>), 4.24 (s, 2H, CH<sub>2</sub>), 6.88 (d, 2H, phenyl J = 8.5), 7.19 (d, 2H, phenyl, J = 8.4) 7.31 (m, 2H, benzofuranyl), 7.47 and 7.96 ppm (2m, 2H, benzofuranyl).

Anal. Calcd. for  $C_{20}H_{20}O_3$  (308.38): C, 77.90; H, 6.54. Found: C, 77.64; H, 6.72.

## 1-[3-(2-Butylbenzofuranyl)]-2-(4-methoxyphenyl)ethanone (4d).

This compound was obtained from 2c and 3b by Method A in 67% yield as a pale yellow oil, bp 194-196°/1 mm Hg; ir (carbon tetrachloride): v CO 1670-1680 cm<sup>-1</sup>,  $^{1}$ H nmr:  $\delta$  0.94 (t, 3H, CH<sub>3</sub>, J = 7.4), 1.40 (m, 2H, CH<sub>2</sub>), 1.74 (m, 2H, CH<sub>2</sub>), 3.15 (t, 2H, CH<sub>2</sub>, J = 7.6), 3.79 (s, 3H, OCH<sub>3</sub>), 4.24 (s, 2H, CH<sub>2</sub>), 6.88 (d, 2H, phenyl, J = 8.5), 7.16 (d, 2H, phenyl, J = 8.5), 7.32 (m, 2H, benzofuranyl), 7.47 and 7.96 ppm (2 m, 2H, benzofuranyl).

*Anal.* Calcd. for C<sub>21</sub>H<sub>22</sub>O<sub>3</sub> (322.40): C, 78.23; H, 6.88. Found: C, 78.20; H, 6.97.

## 1-[3-(2-Ethylbenzofuranyl)]-2-(4-hydroxyphenyl)ethanone (5a).

A mixture of 4b (2.9 g, 0.01 mole), and pyridine hydrochloride (11.5 g, 0.1 mole) was shaking and heated under reflux for 7-9 minutes. The warm solution was poured into 100 ml of ice water, and mixture was allowed to stand for one hour. The aqueous solution was decanted and the residue dissolved in 20 ml of benzene. The benzene solution was washed with 5% hydrochloric acid, water and extracted twice with 5% sodium hydroxide. Then the alkaline solution was acidified with 5% hydrochloric acid the precipitate was filtered and crystallized from methanol to give colorless needles, 2.2 g (80%), mp 123-124°; ir (chloroform): v 1670 (C=O), 3300-3400 (OH) cm<sup>-1</sup>; <sup>1</sup>H nmr: δ 1.32 (t, J = 7.5, 3H, CH<sub>3</sub>), 3.19 (q, J = 7.5, 2H, CH<sub>2</sub>), 4.22 (s, 2H, CH<sub>2</sub>), 5.79 (broad s, OH) 6.74 (dd, J = 8.4, 2H, phenyl), 7.07 (dd, J = 8.4, 2H, phenyl), 7.32 (m, 2H, benzofuranyl), 7.48 and 7.96 (2m, 2H, benzofuranyl).

Anal. Calcd. for  $C_{18}H_{16}O_3$  (280.32): C, 77.12; H, 5.75. Found: C, 77.03; H, 5.90.

1-[3-(2-Propylbenzofuranyl)]-2-(4-hydroxyphenyl)ethanone (5b).

In like manner as for 5a, from the reaction of 4c (3.0 g, 0.01 mole) and pyridine hydrochloride (11.5 g, 0.1 mole) for 10 minutes, 5b (2.0 g, 70%) was obtained as white crystals mp 85-86°, after crystallization from hexane; ir (chloroform): v 1670 (C=O), 3300-3400 (OH) cm<sup>-1</sup>;  $^{1}$ H nmr:  $\delta$  1.02 (t, 3H, CH<sub>3</sub>, J = 7.4), 1.76 (sextet, 2H, CH<sub>2</sub>, J = 7.4), 3.15 (t, 2H, CH<sub>2</sub>, J = 7.6), 4.23 (s, 2H, CH<sub>2</sub>), 5.78 (broad s, OH) 6.88 (d, 2H, phenyl J = 8.5), 7.18 (d, 2H, phenyl, J = 8.4) 7.32 (m, 2H, benzofuranyl), 7.47 and 7.96 ppm (2m, 2H, benzofuranyl).

*Anal.* Calcd. for C<sub>19</sub>H<sub>18</sub>O<sub>3</sub> (294.35): C, 77.53; H, 6.16. Found: C, 77.32, H, 6.32.

## 1-[3-(2-Butylbenzofuranyl)]-2-(4-hydroxyphenyl)ethanone (5c).

This compound was prepared in a similar manner as for 5b from 3.2 g (0.01 mole) of 4d and 11.5 g (0.1 mole) pyridine hydrochloride for 12 minutes. White solid compound 5c had mp 57-58° (2.3.g, 75%); ir (chloroform): v 1670 (CO), 3300-3350 (OH) cm<sup>-1</sup>;  $^{1}$ H nmr:  $\delta$  0.85 (t, J = 7.5, 3H, CH<sub>3</sub>), 1.32 (m, 2H, CH<sub>2</sub>), 1.66 (m, 2H, CH<sub>2</sub>), 3.07 (t, J = 7.6, 2H, CH<sub>2</sub>), 4.14 (s, 2H,

CH<sub>2</sub>), 5.77 (broad s, OH), 6.66 ppm (dd, J = 8.4, 2H, phenyl) 6.99 (dd, J = 8.4, 2H, phenyl), 7.24 (m, 2H, benzofuranyl), 7.39 and 7.86 (2m, 2H, benzofuranyl).

Anal. Calcd. for  $C_{21}H_{20}O_3$  (320.39): C, 78.73, H, 6.29. Found: C, 78.60; H, 6.40.

1-[3-(2-Butylbenzofuranyl)]-2-(3-iodo-4-methoxyphenyl)-ethanone 6.

To a stirred solution of 4d (1.5 g, 0.005 mole) and iodine (2.5 g, 0.01 mole) in 10 ml of ethanol (15 ml) was added concentrated sulfuric acid (0.4 ml). The solution was heated to 60° and then 30% hydrogen peroxide solution (1.5 ml) was slowly added. Under these conditions, the reaction mixture foamed and precipitation occurred. The reaction mixture was stirred for 1 hour, cooled and a dark brown solid was collected. The solid was crystallized from ethanol to yield 1.1 g (51%), mp 109-110°,  $^{1}$ H nmr (deuteriochloroform):  $\delta$  0.94 (t, J = 7.5, 3H, CH<sub>3</sub>), 1.42 (m, 2H, CH<sub>2</sub>), 1.76 (m, 2H, CH<sub>2</sub>), 3.15 (t, J = 7.5, 2H, CH<sub>2</sub>), 3.87 (s, 3H, OCH<sub>3</sub>), 4.20 (s, 2H, CH<sub>2</sub>CO), 6.80 (d, J = 8.2, 1H, phenyl), 7.20 (dd, J = 8.0, 1H, phenyl), 7.33 (m, 2H, benzofuranyl), 7.49 (m, 1H, benzofuranyl), 7.67 (as, 1H, I-phenyl), 7.93 (m, 1H, benzofuranyl).

Anal. Calcd. for C<sub>21</sub>H<sub>21</sub>O<sub>2</sub>I (432.30); C, 58.35; H, 4.90; I, 29.36. Found: C, 58.20; H, 5.03; I, 29.15.

1-[3-(2-Ethylbenzofuranyl)]-2-(3,5-diiodo-4-hydroxy)ethanone 7.

This compound was obtained from 5a (1.4 g, 0.005 mole) in ethanol (10 ml) by proceeding in the same manner as for 6, with the exception that an apparatus with large dimensions and a lower temperature (50°) had to be used because the oxidation of hydroiodide by hydrogen peroxide solution is violent. After adding the hydrogen peroxide solution, the mixture was stirred for 15 minutes, cooled and the yellow precipitate was crystallized from ethanol, yield 0.9 g (35%) light beige, mp 178-179°;

<sup>1</sup>H nmr (deuteriochloroform): δ 1.37 (t, J = 7.5, 3H, CH<sub>3</sub>), 3.20 (q, J = 7.3, 2H, CH<sub>2</sub>), 4.16 (s, 2H, CH<sub>2</sub>CO), 5.73 (bs, 1H, OH) 7.35 (m, 2H, benzofuranyl), 7.50 (dd, 1H, benzofuranyl), 7.58 (s, 2H, 1-phenyl), 7.90 ppm (dd, 1H, benzofuranyl).

Anal. Calcd. for  $C_{19}H_{14}O_{2}I_{2}$  (528.13): C, 43.21; H, 2.67; I, 48.06. Found: C, 43.0; H, 2.82; I, 47.78.

#### REFERENCES AND NOTES

- [1] R. Charlier, G. Deltour, R. Tondeur and F. Binon, Arch. Intem. Pharmacodyn., 139, 234 (1962); Chem. Abstr., 58, 3788c (1963).
- [2] P. Tonboul, G. Kirkorian and G. Atallah, Recent Dev. Cardiovascular Drugs, 1982; Chem. Abstr., 98, 10927 (1983).
- [3] M. Descamps, F. Binon and J. van der Elst, Bull. Soc. Chim. Belges, 73, 459 (1970).
- [4] B. Levitt, M. Stolar and R. Breiman, US Patent 4,831,054; 1989 Chem. Abstr.. 111, 84145 (1989).
- [5] H. D. Taylor and L. Bernard, German Patent 2,445,120 (1975); Chem. Abstr., 83, 28086 (1975).
- [6] J. M. Gonzales Bosch and P. J. Gris Seoane, Spanish Patent, ES 520,590 (1985); Chem. Abstr., 107, 58839 (1987).
- [7] J. P. Bachelet and P. Demarieman, Eur. J. Med. Chim.-Chim. Ther., 17, 323 (1982).
- [8] G. Somari and M. A. Kumar, J. Agric. Food Chem., 32, 762 (1984); Chem. Abstr., 101, 50130s (1984).
  - [9] A. Areschka and F. Binon, Ind. Chim. Belg., 37, 89 (1972).
  - [10] Organic Synthesis, 46, 28 (1966).
  - [11] H. Kwiecień, Pol. J. Chem., 67, 661 (1993).
  - [12] H. Kwiecień, Pol. J. Chem., 70, 733, (1996).
- [13] R. Royer and P. Demersemann, Bull. Soc. Chim. France, 1026 (1968).
- [14] R. Rene, J. P. Buisson and R. Royer; Bull. Soc. Chim. France, 2763 (1975).
  - [15] Sosa, Ann. Chim., 11, 14 (1940); Beilstain E III 10, 434.