SYNTHESES AND CHIROPTICAL PROPERTIES OF SOME NEW N-(5-BENZOFURAZANOYL)-L-α-AMINO ACIDS AND ESTERS

Sadiq A. Saleh*
Chemistry Department, Yarmouk University, Irbid, Jordan
Mustafa M. El-Abadelah
Chemistry Department, Jordan University, Amman, Jordan

Abstract ——— A series of new N-(5-benzofurazanoyl)-L-α-amino acids (Ia) and esters (Ib) have been prepared. The cd spectra of the L-aliphatic and L-aromatic amino acid derivatives (Ia) display, in organic solvents, sign inversion for the measured Cotton effect (CE) bands. Comparable trend is also observed for the respective amino ester analogues (Ib). This chiroptical behaviour might be attributed to differences in conformational equilibria of either series. ^{1}H-Nmr and ms spectra of the title compounds are discussed.

INTRODUCTION

Recently we have reported on the syntheses and chiroptical properties of a series of N-(5(6)-benzofuroxanoyl)-L-α-amino acids and esters[1] (II, Scheme 1). L-Aliphatic and L-aromatic derivatives of II exhibited circular dichroism (cd) curves that displayed sign-reversal for certain Cotton effect (CE) bands[1]. In this context, cd data on the "non-oxygenated" benzofurazan chromophore are desirable for comparison. Such studies are hitherto not reported, and might as well shed light on the contribution of the N-oxide function, if any, to the observed[1] chiroptical behaviour of compounds II. This prompted us to investigate the chiroptical behaviour of model N-(5-benzofurazanoyal)-L-α-amino acids and esters (I, Scheme 1). The present work describes the syntheses and spectral properties of compounds I.

RESULTS AND DISCUSSION

A. SYNTHESES

The N-(5-benzofurazanoyl)-L-α-amino acids (I, 6a-10a, Scheme 1 and Table 1) were obtained by direct benzofurazanoylation of the appropriate L-α-amino acid using 5-benzofurazonoyl chloride 1 in a basic medium (10% aqueous sodium carbonate, or 2% aqueous sodium hydroxide). The required acid chloride 1 was prepared by the action of thionyl chioride onto the corresponding acid (2), following standard procedure[2]. The parent acid (2) was in turn obtained by saponification of the corresponding ethyl ester (3)[3]. The latter ester was produced by deoxygenation of the N-oxygenated acid 5a with triethyl phosphite in absolute ethanol under reflux[3]. The acid 2 was identical with an authentic sample obtained by mild oxidation of 5-formylbenzofurazan[4], and was further characterized by conversion to the corresponding methyl ester[4] upon treatment with diazomethane.

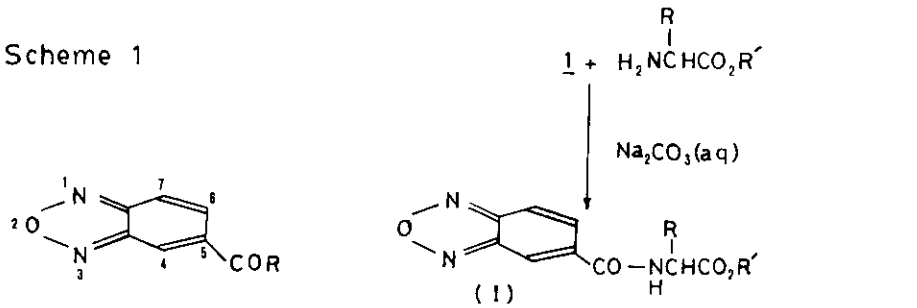
The corresponding methyl esters(I, 6b-10b, Scheme 1) were prepared either by treatment of the parent acids 6a-10a with diazomethane, or by interaction between 5-benzofurazanoyl chloride 1 and the appropriate L-α-amino ester hydrochloride in 10% aqueous sodium carbonate following analogous literature procedure[1]. Compound 10b was also prepared, though less conveniently, by direct coupling between the acid 2 and L-phenylalanine methyl ester hydrochloride using diphenylphosphoryl azide (DPPA) as the coupling reagent[5]. The physical data for compounds 6-10 are given in Table 1.

The reaction conditions employed to prepared series I are quite mild so that the center of chirality is expected to be unaffected; hence compounds I are presumed to be optically pure. This is inferred from previous studies indicating that comparable acylation of the amino function, under similar conditions, led to no detectable racemization[1,6].

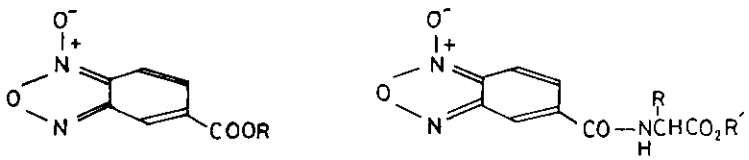
B. SPECTRAL DATA

Mass Spectra: The ms spectra of compounds 1-10 show the correct molecular ions expected for the molecular formulae. In both acid 6a-10a and ester 6b-10b derivatives, bond cleavage at the carboxy grouping produces the aldiminium ion IV ($>$ 50%), possibly via the acylium ion III ($<$ 2%) (Scheme 2). Also, the amide cleavage predominates to give the benzofurazanoylium ion V as the base peak except for 10a (65%) and 10b (70%) where the base peaks are at m/z 148 and 162,

Scheme 1



No.	R	No.	R	No.	R
1	Cl	6	H	9	Ph
2	OH	7	Me	10	CH_2Ph
3	OEt	8	$CHMe_2$		
4	OMe	(a:R´=H ; b:R´=Me)			



5a(R = H)

5b(R = Et)

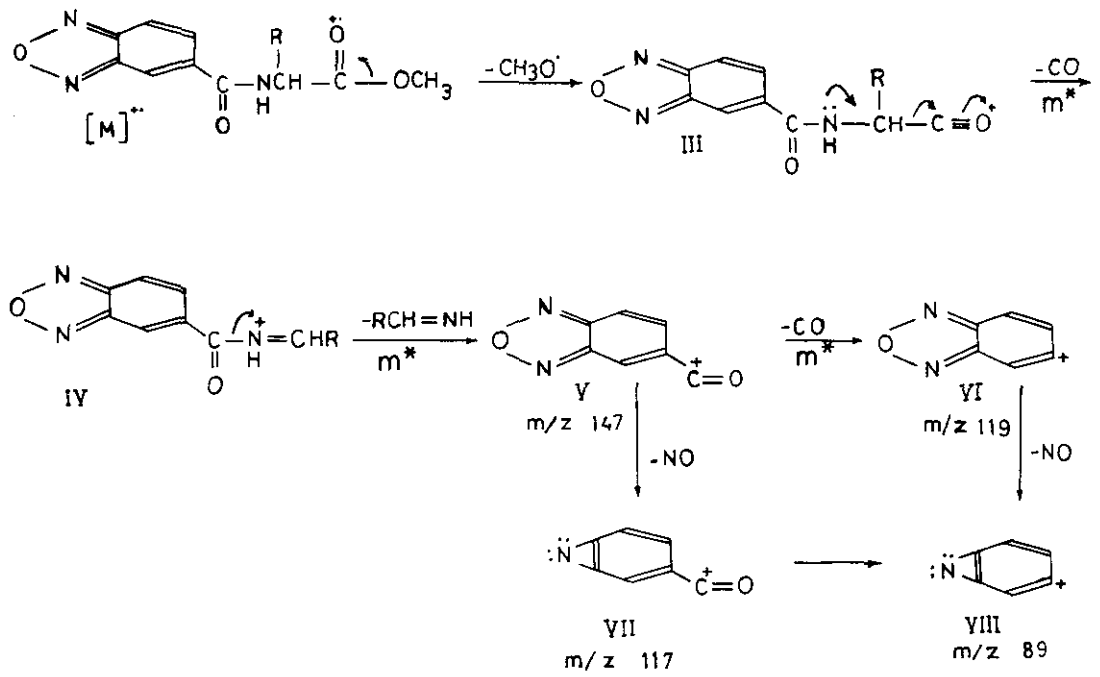
respectively, a result of the competing McLafferty rearrangement process. The latter rearrangement seems to be less important in the valine derivatives 8a and 8b. The ion III undergoes further fragmentation either via loss of carbon monoxide or, more favourably, via extrusion of nitric oxide (NO) to furnish VI (4-7%) and VII (12-20%), respectively. From either of these ions, the nitrenium ion VIII (possibly in equilibrium with its azepinium ion) is then generated in 8-13% relative abundance.

It is worthnoting that direct loss of nitric oxide from the molecular ion is not observed in these amide derivatives 6-10. This process becomes important only after the amide cleavage has proceeded. However, the $[M-NO]^+$ peak is observed

Table 1. Analyses and Physical Data of Compounds I (6-10).

Compd.	Mp(°C)	Mol. Formula	% Analyses Calcd C	Calcd H	Calcd N	Found C	Found H	Found N
6a	174-175	$C_9H_7N_3O_4$	48.88	3.19	19.00	48.62	3.24	18.91
6b	133-134	$C_{10}H_9N_3O_4$	51.07	3.86	17.87	50.83	3.87	17.87
L-7a	139-140	$C_{10}H_9N_3O_4$	51.07	3.86	17.87	50.68	3.92	17.69
L-7b	122-123	$C_{11}H_{11}N_3O_4$	53.01	4.45	16.86	53.12	4.46	16.83
L-8a	155-156	$C_{12}H_{13}N_3O_4$	54.75	4.98	15.96	54.48	5.04	15.75
L-8b	79-80	$C_{13}H_{15}N_3O_4$	56.31	5.45	15.15	56.18	5.39	15.06
L-9a	159-160	$C_{15}H_{11}N_3O_4$	60.61	3.73	14.14	60.29	3.80	13.95
L-9b	109-110	$C_{16}H_{13}N_3O_4$	61.73	4.21	13.50	61.64	4.20	13.45
L-10a	172-173	$C_{16}H_{13}N_3O_4$	61.73	4.21	13.50	61.74	4.19	13.41
L-10b	127-128	$C_{17}H_{15}N_3O_4$	62.76	4.65	12.92	62.53	4.61	12.89

Scheme 2



in the ms spectra of the parent acid 2 (25%) and the corresponding esters 3 and 4 (~2%), but is absent in the ms of the acid chloride 1. In this context, extrusion of nitric oxide from furazans and benzofurazan has recently been reported[7] as the most important fragmentation mode of the molecular ion. This primary step also prevails in the 5-chloro- and 5-methoxylbenzofurazans, whereas it is quite unimportant in the 4- and 5-methylbenzofurazans[8].

^{1}H-Nmr Spectra. The multiplicity-splitting pattern of the three protons (belonging to the benzofurazan moiety) in the ^{1}H-nmr spectra of compounds 6-10 corresponds to an ABX system, of which H-4 represents the X portion. The latter proton's signal, in hexadeuterioacetone, is centered at δ 8.53 for the aliphatic acid derivatives 6a-8a, whereas it is upfield shifted to δ 8.42 for the aromatic counterpart 10a. Comparable upfield shift is also observed, in the solvent deuteriochloroform, for the aromatic ester 10b as contrasted to the aliphatic ester analogues (δ 8.24 for 6b-8b, and δ 8.14 for 10b). This shielding effect is probably the result of special "interaction" between the phenyl ring and the benzofurazan hetero-ring in 10, a factor absent in the aliphatic series 6-8.

The ^{1}H-nmr spectra of 6-10 also display protons' signals that conform with the different individual amino acid/ester residues.

Uv Spectra (Table 2 and Figure 1). The electronic absorption spectra for solutions of benzofurazan were recorded in the literature[2,9]. Benzofurazan exhibits, in the solvent ethanol, absorption band around 268 nm (ε=3400) ascribed to $n \longrightarrow \pi^*$ electronic transition[9]. In compounds I the uv bands of the absorbing 5-benzofurazanoyl chromophore experience bathochromic shifts as a result of the conjugative effect of the amide carbonyl. Thus, the uv spectra of the derivatized amino acids 6a-10a and the respective esters 6b-10b exhibit, in organic solvents, a strong band at about 206 nm, and two medium bands closely located at ca. 285 and 295 nm whose electronic transitions are probably of $\pi \longrightarrow \pi^*$ origin. A low intensity band in the range 310-320 nm is also observed as a shoulder along the rising tail of the medium bands, and is probably of $n \longrightarrow \pi^*$ origin.

Circular Dichroism Spectra (Table 2 and Figure 1). The cd spectra of N-(5-benzofurazanoyl) L-α-alanine, and -L-α-valine (7a, 8a) display, in methanol, positive Cotton effect bands at ca. 285 and 295 nm that coincide with uv absorption maxima. Two stronger CE bands of opposite signs at ca. 250 and 217 nm (negative and positive, respectively) are also recorded; the former band coincides with a uv minimum, indicating that its electronic transition is probably magnetic-dipole allowed, but electric-dipole forbidden in the zero order. The $n \longrightarrow \pi^*$ electronic transition is also optically active showing a positive CE maximum at about 310 nm (shoulder), albeit with a low value for the differential dichroic absorption ($\Delta\varepsilon$). The conjugation bands at 285 and 295 nm show a two-fold increase in the value of $\Delta\varepsilon$ (hyperchromic effect) when goining from methanol to acetonitrile or dioxane, while the shoulder located at longest wavelength is red-shifted. Similar cd trend is also observed for the corresponding esters (7b, 8b) of this L-α-aliphatic series.

The cd spectra of the aromatic L-α-phenylalanine and L-phenylglycine derivatives (9a, 10a) in methanol are similar, but differ from those of the aliphatic L-α-amino acid counterparts (7a, 8a) in the signs of the CE bands. In the former series, sign inversion is observed for the CE bands located at longest wavelengths (positive), as well as the CE band at ca. 215 nm (negative). Comparable cd trend is likewise observed in the solvents acetonitrile and dioxane, specially

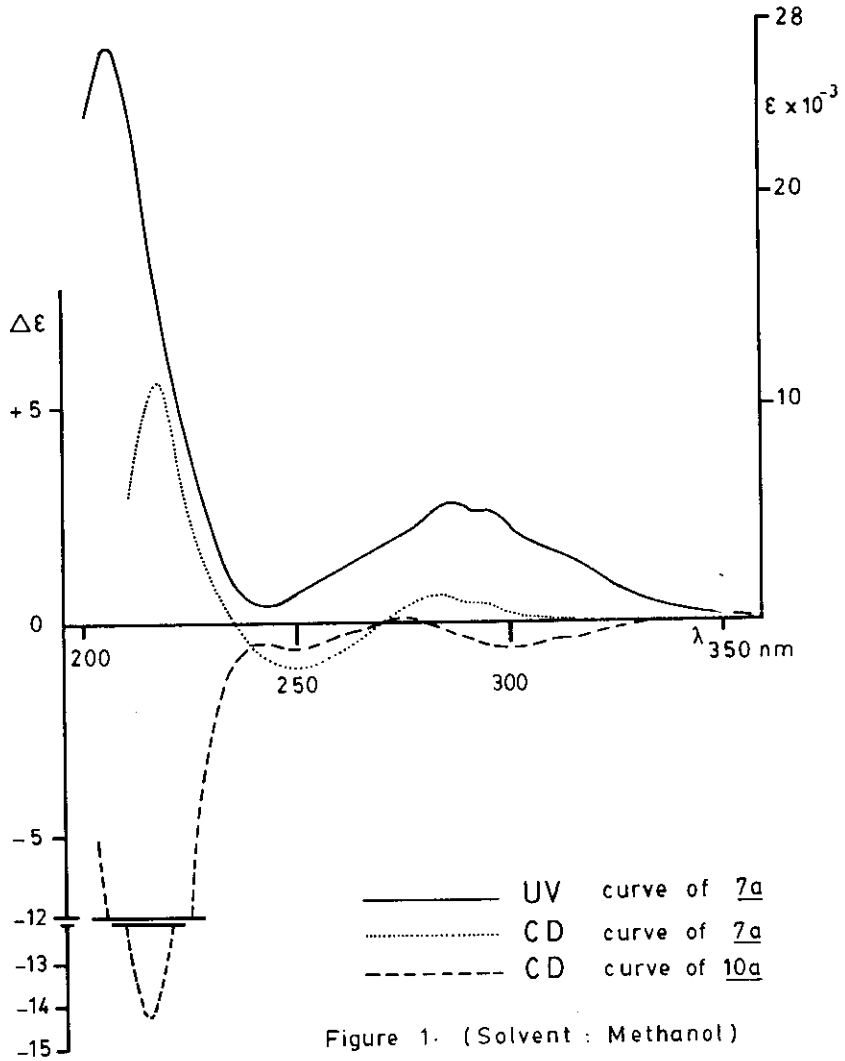


Figure 1. (Solvent : Methanol)

for the intense highest energy band around 215 nm which is invariably negative. Corresponding cd behaviour is also observed for the L-aromatic amino ester analogues (9b, 10b). This phenomenon of sign reversal was previously observed for the L-aliphatic and L-aromatic series of the respective N-oxygenated compounds (II)[1], and is probably the result of differences in relative population of conformational isomers in either series. Arguments along the lines adopted in previous, related work[1] apply also here. Thus, it is assumed that in the

Table 2. CD and UV Spectral Data of the L-Derivatives 6-10.

No.	Solvent[c]	CD[d] / λ_{max} ($\Delta\varepsilon$)	UV[d] / λ_{max} ($\varepsilon \times 10^{-3}$)
7a	M	308sh(+0.08), 295(+0.41), 284(+0.66), 250(-1.13), 217(+5.65).	310sh(3.3), 295(5.1), 285(5.4), 275sh(4.8), 206(27.0).
	A	326(-0.07), 295(+1.10), 286(+1.36), 252(-1.25), 217(+5.29).	315sh(2.7), 295(4.8), 285(5.1), 274sh(4.4), 207(23.8).
	D	330(-0.05), 295(+1.05), 285(+1.20), 253(-1.10), 217(+5.58).	315sh(3.3), 295(5.3), 286(5.5), 278sh(5.1), 206(26.9).
8a	M	325(-0.05), 295(+0.44), 285(+0.63), 250(-0.80), 220(+3.97).	320sh(2.3), 295(5.0), 285(5.4), 275sh(4.8), 206(25.8).
	A	325(-0.12), 295(+1.62), 285(+1.97), 253(-1.15), 222(+7.53).	317sh(2.9), 297(5.3), 285(5.6), 275sh(5.0), 206(30.3).
	D	328(-0.06), 297(+1.35), 288(+1.48), 254(-1.00), 222(+4.51).	315sh(3.1), 296(5.1), 287(5.3), 276sh(4.7), 207(22.6).
9a	M	310(-0.20), 297(-0.36), 288(-0.32), 264(+0.21), 226(+3.10), ~210(-ve)[e].	310sh(3.7), 295(5.5), 285(5.9), 274sh(5.3), 265sh(4.4), 258sh(4.0), 206(35.8).
	A	311(-0.11), 274(+0.20), 265(+0.16), 258(+0.11), 226(+3.90), ~210(-ve)[e].	311sh(3.1), 296(5.1), 285(5.4), 275sh(4.9), 265sh(4.1), 258sh(3.5), 206(35.3).
	D	313(-0.20), 275(+0.28), 265(+0.20), 227(+3.85), ~212(-ve)[e].	314sh(3.0), 296(4.9), 287(5.1), 276sh(4.5), 265sh(3.9), 258sh(3.4), 206(35.9).

10a	M	315(-0.40), 302(-0.54), 275(+0.12), 265(-0.13), 250(-0.63), 215(-14.25).	315sh(2.7), 295(5.0), 285(5.3), 275sh(4.7), 265sh(3.8), 206(34.0).
	A	316(-0.38), 283(+0.70), 266sh(-0.25), 253(-1.00), 213(-15.36).	315sh(2.8), 295(5.1), 285(5.4), 276sh(4.9), 265sh(3.9), 258sh(3.2), 207(39.1).
	D	318(-0.30), 280(+0.57), 273(+0.48), 250(-0.23), 216(-6.35).	315sh(2.7), 297(4.4), 287(4.5), 279sh(4.2), 260sh(2.9), 206(32.0).
7b	M	327(-0.02), 295(+0.60), 284(+0.84), 276sh(+0.68), 250(-1.34), 215(+6.08).	315sh(3.7), 295(5.0), 284(5.4), 275sh(4.9), 207(27.3).
	A	325(-0.06), 295(+1.10), 285(+1.30), 275sh(+0.65), 252(-1.28), 216(+6.17).	311sh(3.2), 296(5.0), 285(5.3), 274sh(4.6), 207(27.5).
	D	330(-0.06), 295(+1.30), 287(+1.38), 277sh(+0.76), 253(-1.21), 215(+6.30).	315sh(3.3), 296(5.1), 286(5.2), 275sh(4.6), 207(25.3).
8b	M	323(-0.03), 295(+0.60), 283(+0.90), 276sh(+0.58), 250(-1.00), 220(+4.00).	318sh(2.5), 295(4.7), 284(5.1), 275sh(4.5), 206(23.3).
	A	325(-0.11), 296(+1.45), 286(+1.81), 275sh(+1.05), 252(-1.26), 220(+6.34).	320sh(2.3), 295(5.5), 285(5.8), 275sh(5.2), 206(27.3).
	D	330(-0.05), 296(+1.37), 288(+1.55), 275sh(+0.96), 250(-1.02), 223(+5.04).	315sh(2.7), 296(4.4), 287(4.6), 275sh(4.0), 206(23.4).
9b	M	310sh(-0.27), 296(-0.51), 287(-0.38), 270(+0.34), 265(+0.40), 258(+0.31), 224(+5.86), ~210(-ve)[e].	310sh(2.8), 295(4.4), 285(4.8), 273sh(4.2), 265sh(3.6), 258sh(3.1), 205(32.6).

	A	311(-0.28), 275(+0.40), 265(+0.30), 258(+0.13), 225(+6.35), ~210(-ve)[e].	312sh(2.9), 296(4.5), 285(4.9), 273sh(4.4), 265sh(3.7), 258sh(3.3), 207(33.8).
	D	325sh(-0.21), 315(-0.28), 278(+0.70), 266(+0.35), 255(-0.22), 250(-0.30), 226(+6.00), ~212(-ve)[e].	315sh(2.6), 296(4.3), 286(4.6), 277sh(4.2), 265sh(3.5), 260sh(3.2), 254sh(2.7), 206(28.7).
10b	M	312sh(-0.30), 302(-0.40), 302(-0.44), 277(+0.07), 250(-1.08), 215(-14.72).	310sh(3.3), 295(5.3), 285(5.7), 277sh(5.2), 265sh(4.1), 258sh(3.6), 206(34.0).
	A	316(-0.31), 295sh(+0.28), 284(+0.61), 253(-0.86), 215(-11.50).	315sh(2.7), 295(4.7), 285(5.0), 275sh(4.4), 264sh(3.5), 258sh(2.9), 252sh(2.4), 207(31.6).
	D	320(-0.26), 283(+0.64), 250(-0.42), 216(-5.36).	317sh(2.6), 295(4.4), 287(4.6), 280sh(4.3), 275sh(4.1), 265sh(3.4), 260sh(2.9), 255sh(2.6), 207(30.1).
6b	M		310sh(3.1), 295(5.0), 285(5.3), 275sh(4.7), 207(24.6).
	A		312sh(3.3), 296(4.9), 285(5.2), 275sh(4.6), 207(27.7).
	D		313sh(3.4), 295(5.0), 286(5.1), 278sh(4.8), 207(27.0).

[c]Solvents used: M = Methanol ; A = Acetonitrile ; D = Dioxane.

[d]sh = Shoulder. [e]$\Delta\varepsilon$ value could not be determined with certainty.

L-aromatic series (9, 10) a conformation predominates that allows "interaction" between the phenyl moiety of the amino acid residue and the benzofurazan chromophore. This aryl-heteroaryl "interaction" is manifested in the ^{1}H-nmr spectral data; the benzofurazanoyl protons' signal at lowest field is upfield shifted in 10 relative to an invariant position in the aliphatic analogues 6-8 (vide supra).

In conclusion, sign inversion of certain CE bands, formerly reported for the benzofurazan N-oxides (II)[1], is also recorded in this study for the non-oxygenated benzofurazan analogues (I). Apparently, the origin of this phenomenon appears to be governed largely by conformational factors that allow differences in "interaction" between the hetero-ring and the L-aliphatic/L-aromatic amino acid residues. In these systems, the N-oxide function contributes little, if any, to the observed chiroptical behaviour. In contrast, the N-oxidation effect plays a dominant role that explains differences in the chiroptical properties of the N-(2-quinoxaloyl)-L-α-amino acids as compared to the corresponding quinoxaline N-oxides[10].

EXPERIMENTAL

L-α-amino acids and the corresponding esters were of Biochemical grade (Fluka). Merck silica gel ($HF_{254+360}$) was used for preparative tlc plates, with chloroform as the eluent.

Melting points were determined in capillary tubes on a Gallenkamp apparatus and are uncorrected. Optical rotation measurements were obtained with a Perkin-Elmer 141 polarimeter in dimethylformamide for the acids, and in chloroform for the esters (c, 1-2). ^{1}H-Nmr spectra were recorded on JEOL - MH - 100 (100 MHz) or a Brucker-FX-90 (90 MHz) spectrometers using deuteriochloroform, or hexadeuterioacetone as solvents and tetramethylsilane as an internal standard. Uv spectra were run with a Cary-17 spectrophotometer in cells of 0.01-0.05 cm pathlength. Cd spectra were recorded on a Jobin Yvon Dichrograph III Division d'instruments S.A for solutions in methanol, acetonitrile and dioxane at 20°C in cells of 0.01-0.2 cm path length; concentrations were in the range of 0.2-0.8 mg/ml. Mass spectra were obtained with a Varian CH-5 spectrometer (70 eV) using the direct inlet technique.

Microanalyses were carried out at the laboratories of Dr. F. Pascher/E. Pascher (Bonn-West Germany), and at UMIST (England).

General Procedures.

5-Ethoxycarbonylbenzofurazan (3). 5(6)-Carboxybenzofuroxan[2] (5a , 0.025 mol) and triethyl phosphite (20 ml) in absolute ethanol (200 ml) were refluxed for 3 h. The alcohol was evaported in vacuo (120 mmHg); the residue was then steam-distilled, and the product was collected from the cooled distillate. Yield = 78%; mp = 41-42°C (Lit[3]. mp = 42-43°C).

5-Carboxybenzofurazan (2). Compound 3 (0.02 mol) in aqueous sodium hydroxide (5%, 80 ml) was heated on a water bath for 30-40 min. The resulting clear solution was filtered, the cooled filtrate was acidified with 5N hydrochloric acid to pH 3, and the precipitated product was collected. Yield = 90%; mp=160-161°C (Lit[4]. mp = 159°C).

5-Methoxycarbonylbenzofurazan (4). This ester was obtained in almost quantitative yield by treatment of the parent acid 2 with diazomethane in ether,and was purified by vacuum sublimation (95°C/30 mm Hg). Mp=44-45°C. Anal. Calcd for $C_8H_6N_2O_3$:C,53.94; H,3.93; N,15.72. Found: C,54.08 ; H,3.45 ; N,15.56.

5-Benzofurazanoyl Chloride (1). The acid 2 (0.02 mol) and thionyl chloride (12 ml) in toluene (40 ml) were refluxed for 3 h. Excess of thionyl chloride and toluene were removed in vacuo, and the oily residue was crystallized from petroleum ether as fine colourless needles. Yield =80-85%; mp =51-52°C. Anal. Calcd for $C_7H_3N_2O_2Cl$: C,46.05; H,1.66; N,15.34; Cl,19.42. Found: C,45.82 ; H,1.59; N,15,27; Cl,19.36.

N-(5-Benzofurazanoyl)-α-amino Acids (6a-10a, Table I). The acid chloride (1 , 0.01 mole) was added, over 30 min , to a stirred solution of the particular amino acid (0.02 mol) in aqueous sodium hydroxide (5%, 25 ml) or sodium carbonate (10%, 20 ml) at 0-5°C. The resulting violet-coloured solution was further stirred at ambient temperature until a yellow colouration was acquired (2-4 h.). The reaction mixture was then filtered, the cooled filtrate was acidified with 5N hydrochloric acid to pH 3, and the precipitated product was collected (compound 6a separated slowly upon cooling for overnight). Yields were in the range of 65-80%.

N-(5-Benzofurazanoyl)-α-amino Acid Methyl Esters (6b-10b, Table 1). (i) The particular acid (0.01 mol) in ether (50 ml) was treated with diazomethane in ether in the conventional manner. The solvent was then evaporated, and the residue was crystallized from the appropriate solvent. Yields were in the range of 80-90%.

(ii) 5-Benzofurazanoyl chloride (1 , 0.01 mol) in tetrahydrofuran (10 ml) was added dropwise to a stirred solution of the appropriate α-amino ester hydrochloride (0.012 mol) in aqueous sodium carbonate (10%, 60 ml) at 2-5°C. The resulting mixture was stirred for additional 20 min , diluted with water (40 ml), and extracted with diethyl ether or chloroform (2x30 ml). The combined organic extracts were dried ($MgSO_4$), the solvent was evaporated, and the residue was purified by recrystallization from a suitable solvent. Yields were in the range 60-75%.

(iii) A solution of the acid (2, 0.01 mol) and DPPA (0.012 mol) in dimethylformamide (25 ml) was stirred at 0-5°C for 30 min. L-α-phenylalanine methyl ester hydrochloride (0.012 mol) and triethyl amine (4 ml) were then introduced, successively; the reaction mixture was further stirred for 2 h at 0°C, and for 6 h at room temperature. The solution was then filtered, the filtrate was diluted with cold water (150 ml), the precipitated product (10b) was collected and purified on preparative tlc plates. Yield = 55%.

ACKNOWLEDGEMENTS

This work was supported by the Research Council-Yarmouk University. We are grateful to Prof. Dr. E. Steiglich (Bonn Universitat, BDR) for Instrumentation facilities.

REFERENCES

1. M.M. El-Abadelah, A.A. Anani, Z.H. Khan,and A.M. Hassan, J. Heterocyclic Chem., 1980, 17, 213.
2. A.J. Boulton, P.B. Ghosh,and A.R. Katritzky, J. Chem. Soc. (C), 1966, 971.
3. P.B. Ghosh and M.W. Whitehouse, J. Med Chem., 1968, 11, 305. These authors reported that deoxygenation of 5a produced the corresponding acid 2 with mp= 42-43°C, and with satisfactory C,H, N analysis (for $C_7H_4N_2O_3$). We have

found that, under identical experimental conditions, the product of deoxygenation of 5a was in fact the deoxygenated ethyl ester 3, but not the acid 2 ; apparently esterification has occurred. The ester 3 was identical with a sample preparation the authors described in a later publication (P.B. Ghosh, B. Ternai, and M.W. Whitehouse, J. Med. Chem., 1972, 15, 255) via analogous deoxygenation of 5b.

4. D. Dal Monte, E. Sandri, and W. Cere', Ann. Chim. (Rome), 1970, 60, 801.
5. T. Shioiri, K. Ninomiya, and S. Yamada, J. Am. Chem. Soc., 1972, 94, 6203.
6. M.M. El-Abadelah, S.S. Sabri, H.D. Tabba, and H. Duddeck, J. Heterocyclic Chem., 1977, 14, 871 ; M.M. El-Abadelah, S.S. Sabri, A.A. Jarrar, and M.H. Abu-Zarga, J. Chem. Soc., Perkin I, 1979, 2881.
7. C.L. Pederson and J. MØller, Acta Chem. Scand., 1975, B29, 483.
8. F. Razmara and E. Waight, Org. Mass Spectrom., 1976, 11, 1227.
9. D. Dal Monte and E. Sandri, Bull. Sci. Fac. Chim. Ind. Bologna, 1964, 22, 33.
10. M.M. El-Abadelah, S.S. Sabri, M.Z. Nazer, and M.F. Za'ater, Tetrahedron, 1976, 32, 2931.

Received, 27th March, 1989