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Synthesis of 4,5,6,7-Tetraphenyl-8-(substituted)-3(2H)-phthalazinone Derivatives Likely to Posses Antihypertensive Activity

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The interaction of tetraphenylphthalic anhydride with *o*-chlorotoluene under Friedel-Craft condition gives 2-(4-chloro-3-methyl)benzoyl-3,4,5,6-tetraphenyl benzoic acid(1), which on reaction with hydrazine derivatives gave phthalazinones (2a-d). The behaviour of (2a) towards carbon electrophiles and carbon nucleophiles has been investigated. The chlorophthalazinones (4a) also has been synthesised from the action of $\text{PCl}_5/\text{POCl}_3$ on (2a). The behaviour of (4a) towards nitrogen, and oxygen nucleophiles also have been described.

Introductions

Recently^{1,2}, 3 (2H)-phthalazinones have been described as being useful as remedies for artroisosclerosis and thromosis, and have also been useful as antihypertensive mate-

rials. This promoted us to synthesis some new phthalazinones.

Thus, interaction of tetraphenylphthalic anhydride with *o*-chlorotoluene under Friedel-Craft condition gives 2-(4-chloro-3-methyl)benzoyl-3,4,5,6-tetraphenyl benzoic acid

(1), which reacts with hydrazines namely, hydrazine hydrate, phenylhydrazine, benzoylhydrazine and salicylhydrazine in boiling butanol to give the corresponding phthalazinone derivatives (2a-d). It was also reacts with semicarbazide and thiosemicarbazide in pyridine to give (2e and 2f).

The structure of compounds (2a-f) was confirmed by: (i) analytical data (ii) IR spectrum of (2a) shows bands at 1640, 1670, 3150 and 3400 attributable to $\nu_{C=O}$, ν_{C-N} , ν_{NH} and ν_{OH} respectively.

The identity of the compound (2c) was deduced vis treatment of (2a) with benzoyl chloride in pyridine.

On the other hand, compound (2a) reacts with a variety of electrophiles namely, dimethyl sulphate, ethyl chloroacetate, and ethyl chloroformate in dry acetone, by using anhydrous K_2CO_3 as catalyst³ to give 3-(methyl, ethylacetate and ethyl carboxy) Phthalazine derivatives (3a-c) respectively.

Structure of compound (3b and c) was established from: (i) IR. Spectra exhibit strong absorptions at 1635, 1740, attributable to $\nu_{C=N}$ and $\nu_{C=O}$ of ester. (ii) chemically, via the reaction of (3b and 3c) with each of: a) aqueous sodium hydroxide to give the corresponding acids (3d and 3e) respectively, b) hydrazine and phenylhydrazine in boiling butanol to give the corresponding hydrazides (3f-i), c) with primary amines namely, aniline, ethylamine, benzylamine and P-anisidine in boiling butanol to give the corresponding N-aryl and N-alkyl amide derivatives (3j-p). The IR spectra of (3f-i) exhibit bands in the region (1660-1665), $\nu_{C=O}$ carboxamide) and (3200-3300) ν_{NH} of two NH groups. Compounds (3j-p) show bands in the region of (1660-1665) and (3200-3300) attributable to $\nu_{C=O}$ and ν_{NH} respectively.

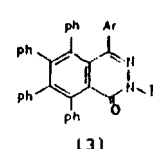
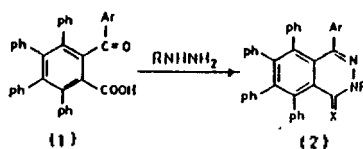
Furthermore, the ester (3b) reacted with benzaldehyde in presence of potassium tert-butoxide at room temperature affording "Stobbe" product (3q) as major product and the "Claisen" product (3r) as minor product⁴. The above ester (3r) was converted to the corresponding acid (3q) by hydrolysis with aqueous NaOH. IR spectrum of (3q) shows bands at 1620, 1635 and 1705 attributable to $\nu_{C=C}$, $\nu_{C=N}$ and $\nu_{C=O}$, respectively.

Also, treatment of (2a) with PCl_5 in presence of $POCl_3$ gave 1-chlorophthalazinone (4a) which its structure was deduced from: (i) Its IR spectrum which shows the disappearance of $\nu_{C=O}$ and ν_{NH} (ii) chemically via reaction of (4a) with nitrogen nucleophiles e.g. hydrazine hydrate, phenylhydrazine, aniline and methylamine affording (hydrazino, phenylhydrazino, anilino and methylamino) phthalazine derivatives (4b-e). Their IR spectra show bands in the region (1630-1625) and (3200-3300) attributable to $\nu_{C=N}$ and ν_{NH} , respectively.

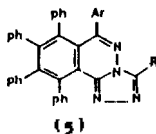
The reaction of the chlorophthalazine derivatives (4a) with acylhydrazines, namely, benzoylhydrazine, phenylacetic-hydrazide and salicylic hydrazide in boiling butanol to give 1,2,4-triazolo-(4,3-b)-4,5-benzopyridazines (5a-c). The IR spectra of (5a-c) revealed bands at the region of 1640-1630 due to $\nu_{C=N}$.

The reaction of phthalazinone (2a) with P_2S_5 in boiling xylene⁵ gives the corresponding phthalazine thione (2g) which IR spectrum revealed bands at 1340 and 3250 attributable to $\nu_{C=S}$ and ν_{S-H} or ν_{NH} .

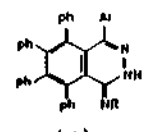
It was reported⁶, that thiopyridazinones under went condensation with nitrogen nucleophiles e.g. hydrazines or pri-



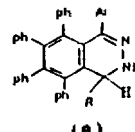
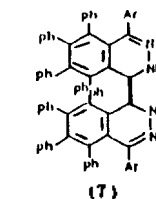
$\text{Ar} = 4,3\text{-Cl}_2\text{C}_6\text{H}_3$



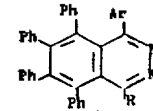
a, R = C_6H_5
b, R = $CH_2C_6H_5$
c, R = $C_6H_4(OH)(O)$



a, R = NH_2
b, R = $NHPh$
c, R = C_6H_5
d, R = CH_3



a, R = CH_3
b, R = C_2H_5



a, R = Cl
b, R = $NHNH_2$
c, R = $NHNHPh$
d, R = $NHPh$
e, R = $NHCH_3$

mary amines to give the corresponding hydrazones and Schiff-bases respectively. In the present work the authors try to compare the behaviour of the thion (2g) quantitatively (no data) towards the nitrogen nucleophiles with the thiopyridazinones. Thus, when (2g) was allowed to react with hydrazine hydrate, phenylhydrazine, aniline and methylamine in boiling ethanol affording (4b-e) and not the corresponding hydrazones or Schiff bases (6a-d), this may be due to compounds (6) produced as fleeting intermediate which rearrange directly to the more thermodynamically stable tautomers (4b-e). Identity of these products were deduced from correct analytical data and from m.p. and m.m.p. determinations.

Treatment of the thione (2g) with copper bronze in boiling xylene yielded the bis-compound (7). Its IR spectrum exhibits bands at 1625 and 1618 attributable to $\nu_{C=N}$ and $\nu_{C=C}$.

Action of Grignard reagents on (2a): when (2a) was submitted to react with methyl magnesium iodide and ethyl magnesium iodide in dry ether-benzene mixture affording (8a and 8b) which its IR spectra show absorption bands in the region 1635-1620 and 3270-3300 attributable to $\nu_{C=N}$ and ν_{NH} respectively.

Experimental

All melting points are uncorrected. IR spectra were determined with a Unicam SP 1200 spectrophotometer using

* max expressed by cm^{-1} throughout the paper.

Table 1. Characterisation and Physical Data for the Synthesised Compounds

Compd.	M.P. °C	Solvent Yield %	Mol. formula (M. wt)	Analysis	
				Calcd.	Found
2a	240	Toluene (65)	C ₃₉ H ₂₇ ClN ₂ O (574.5)	C 81.32	81.02
				H 4.69	4.34
				N 4.86	4.71
2b	135	Benzene (63)	C ₄₅ H ₃₁ ClN ₂ O (650.5)	C 83.01	82.71
				H 4.76	4.52
				N 4.30	4.00
2c	205	Benzene (60)	C ₄₆ H ₃₁ ClN ₂ O ₂ (678.5)	C 81.35	81.00
				H 4.56	4.41
				N 4.12	4.01
2d	230	Ethanol (60)	C ₄₆ H ₃₁ ClN ₂ O ₃ (694.5)	C 79.48	79.32
				H 4.46	4.35
				N 4.03	4.00
2e	260	Benzene (70)	C ₄₀ H ₂₈ ClN ₃ O ₂ (617.5)	C 77.73	77.43
				H 4.53	4.42
				N 6.80	6.51
2f	258	Toluene (60)	C ₄₀ H ₂₈ ClN ₃ OS (633.5)	C 75.76	75.54
				H 4.41	4.32
				N 6.62	6.33
2g	230	Xylene (63)	C ₃₉ H ₂₇ ClN ₂ S (590.5)	C 79.25	79.00
				H 4.57	4.48
				N 4.74	4.65
3a	131	Benzene (55)	C ₄₀ H ₂₉ ClN ₂ O (588.5)	C 81.56	81.46
				H 4.92	4.82
				N 4.75	4.65
3b	115	Light Petrol. 80-100° (65)	C ₄₃ H ₃₃ ClN ₂ O ₃ (660.5)	C 78.12	78.01
				H 4.99	4.87
				N 4.23	4.18
3c	110	Light Petrol. 80-100° (60)	C ₄₂ H ₃₁ ClN ₂ O ₃ (646.5)	C 77.95	77.84
				H 4.79	4.62
				N 4.33	4.29
3d	240	Ethanol (50)	C ₄₁ H ₂₉ ClN ₂ O ₃ (632.5)	C 77.78	77.68
				H 4.58	4.43
				N 4.42	4.40
3e	245	Ethanol (50)	C ₄₀ H ₂₇ ClN ₂ O ₃ (618.5)	C 77.60	77.43
				H 4.36	4.23
				N 4.52	4.43
3f	235	Ethanol (55)	C ₄₁ H ₃₇ ClN ₄ O ₂ (646.5)	C 76.01	75.98
				H 4.79	4.58
				N 8.66	8.53
3g	138	Ethanol (60)	C ₄₇ H ₃₅ ClN ₄ O ₂ (722.5)	C 78.06	78.00
				H 4.84	4.73
				N 7.75	7.63
3h	245	Benzene (65)	C ₄₀ H ₂₉ ClN ₄ O ₂ (632.5)	C 75.88	75.64
				H 4.58	4.48
				N 8.85	8.73
3i	215	Benzene (65)	C ₄₆ H ₃₃ ClN ₃ O ₂ (656.5)	C 84.08	84.00
				H 5.02	4.93
				N 6.39	6.21
3j	131	Toluene (60)	C ₄₇ H ₃₄ ClN ₃ O ₂ (707.5)	C 79.71	79.69
				H 6.08	6.00
				N 4.80	4.72
3k	130	Toluene (65)	C ₄₃ H ₃₄ ClN ₃ O ₂ (659.5)	C 78.24	78.13
				H 6.52	6.48
				N 6.36	6.23
3l	220	Benzene (55)	C ₄₈ H ₃₆ ClN ₃ O ₂ (721.5)	C 79.83	79.77
				H 4.98	4.89
				N 5.82	5.73

3m	206	Toluene (60)	C ₄₆ H ₃₂ ClN ₃ O ₂ (693.5)	C 79.59	79.49
				H 4.61	4.52
				N 6.05	6.00
3n	238	Benzene (60)	C ₄₂ H ₂₈ ClN ₃ O ₂ (641.5)	C 78.56	78.46
				H 4.48	4.43
				N 6.54	6.49
3o	230	Benzene (55)	C ₄₇ H ₃₀ ClN ₃ O ₂ (703.5)	C 80.17	80.00
				H 4.26	4.19
				N 5.97	5.88
3p	205	Benzene (55)	C ₄₇ H ₃₀ ClN ₃ O ₃ (719.5)	C 78.38	78.21
				H 4.16	4.13
				N 5.83	5.79
3q	180	Toluene (55)	C ₄₈ H ₃₃ ClN ₂ O ₃ (720.5)	C 79.94	79.94
				H 4.58	4.49
				N 3.88	3.79
3r	85	Petrol. ether 60-80° (45)	C ₅₀ H ₃₇ ClN ₂ O ₃ (748.5)	C 80.16	80.00
				H 4.94	4.91
				H 3.74	3.69
4a	220	Toluene (60)	C ₃₉ H ₂₆ Cl ₂ N ₂ (592)	C 79.05	78.93
				H 4.39	4.28
				N 4.72	4.69
4b	150	Benzene (55)	C ₃₉ H ₂₉ ClN ₄ (588.5)	C 79.52	79.43
				H 4.92	4.88
				N 9.51	9.49
4c	144	Benzene (60)	C ₄₅ H ₃₃ ClN ₄ (664.5)	C 81.26	81.25
				H 4.96	4.95
				N 8.42	8.36
4d	170	Toluene (63)	C ₄₅ H ₃₂ ClN ₃ (649.5)	C 83.14	82.98
				H 4.92	4.83
				N 6.46	6.39
4e	142	Toluene (65)	C ₄₀ H ₃₀ ClN ₃ (587.5)	C 81.70	81.65
				H 5.10	5.00
				N 7.14	7.01
5a	233	Toluene (50)	C ₄₆ H ₃₁ ClN ₄ (674.5)	C 81.83	81.78
				H 4.59	4.49
				N 8.30	8.00
5b	214	Butanol (60)	C ₄₇ H ₃₃ ClN ₄ (688.5)	C 81.91	81.83
				H 4.79	4.73
				N 8.13	8.12
5c	222	Butanol (60)	C ₄₆ H ₃₁ ClN ₄ O (690.5)	C 79.94	79.90
				H 4.48	4.46
				N 8.10	7.94
7	251	Toluene (60)	C ₇₈ H ₅₄ ClN ₄ (1032.5)	C 90.65	90.00
				H 5.23	5.18
				N 5.42	5.32
8a	225	Toluene (60)	C ₄₁ H ₃₃ ClN ₂ (588.5)	C 83.60	83.51
				H 5.60	5.56
				N 4.75	4.65
8b	205	Toluene (65)	C ₄₃ H ₃₈ ClN ₂ (617.5)	C 83.56	83.50
				H 6.15	6.00
				N 4.53	4.49

KBr Wafer technique. Characterisations and physical data are listed on Table 1.

Formation of Phthalazinone Derivatives (2a-d). A mixture of acid (1) (0.01 mole) and hydrazines namely, hydrazine hydrate, phenylhydrazine, benzoylhydrazine, and salicyloylhydrazine (0.01 mole) in *n*-butanol (50 ml) was refluxed for 3 hrs. The solids separated were filtered off and crystallized from the suitable solvent to give (2a-d).

Formation of Phthalazinone Derivatives (2e and 2f).

A mixture of acid (1) (0.01 mole) and semicarbazide or thiosemicarbazide (0.01 mole) in pyridine (40 ml) was refluxed for 8 hrs. The reaction mixture was poured into ice/HCl (100 gm ice and 20 ml conc. HCl). The solids obtained were filtered off and crystallized from the proper solvent.

Reaction of Phthalazinone (2a) with Benzoyl Chloride: Formation of (2c). A mixture of (2a) (0.01 mole), benzoyl chloride (0.01 mole) and pyridine (1 ml) was refluxed for 3 hrs. The cold reaction mixture was added gradually into ice dilute HCl. The solid obtained was filtered off and crystallized from the proper solvent to give (2c), which was identified by m.p. and m.m.p. determination.

Action of Dimethylsulphate, Ethyl Chloroacetate and Ethyl Chloroformate on Phthalazinone (2a): Formation of Phthalazine Derivatives (3a-c). A mixture of (2a) (0.01 mole), alkylating agent namely, dimethyl sulphate, ethyl chloroacetate, and ethyl chloroformate (0.04 mole); and anhydrous potassium carbonate (0.04 mole) in dry acetone (50 ml) was refluxed for 20 hrs. The excess of solvent was removed by distillation, the reaction mixture was poured into water. The organic material was extracted by ether, evaporating of the dried ethereal solution gave the alkylating products (3a-c) respectively.

Alkaline Hydrolysis of (3b and 3c): Formation of (3d and 3e). A mixture of (3b) or (3c) (0.01 mole) and aqueous sodium hydroxide 10% (10 ml) for each gm of (3b or 3c) was refluxed for 3 hrs. The reaction mixture was cooled and extracted with ether to get red of unsaponified material. The aqueous layer was acidified with very cold dilute hydrochloric acid, the solid obtained was filtered off and recrystallized from the proper solvent to give (3d and 3e).

Action of Hydrazines and Amines on (3b and 3c): Formation of Hydrazides and Amides (3f-p). A mixture of (3b or 3c) (0.01 mole), hydrazine hydrate, phenylhydrazine, aniline, ethyl amine, benzyl amine or *p*-anisidine (0.01 mole) was refluxed in *n*-butanol (50 ml) for 3 hrs. The solid separated after concentrating and cooling was filtered off and recrystallized from the proper solvent.

Action of Benzaldehyde on (3b): Formation of Cinnamic Acid Derivatives (3q and 3r). A mixture of (3b) (0.01 mole) and benzaldehyde (0.01 mole) was added in a course of 30 min to a stirred solution of potassium *tert*-butoxide (in *tert*-butanol, 20 ml for each gm of 3b). The reaction mixture was left overnight at room temperature. The excess of alcohol was removed under reduced pressure. The residue was poured onto water and extracted with ether then, evaporating the dried ethereal layer left on oily material (b.p. 40-60°), which recrystallized from the proper solvent give (3r). The aqueous layer upon acidification with very cold dilute hydrochloric acid gave a precipitate, which filter off and recrystallized from the proper solvent and gave (3g).

Action of PCl_5 / POCl_3 on Phthalazinone Derivative (2a): Formation of Chlorophthalazine Derivative (4a). A mixture of phthalazinone (2a) (0.01 mole), PCl_5 (1 gm) and POCl_3 (7 ml) was refluxed on a steam bath for 3 hrs. The cold reaction mixture was poured slowly into crushed ice. The solid separated was filtered, dried and recrystallized from the

proper solvent to give (4a).

Action of Hydrazines and Amines on Chlorophthalazine (4a): Formation of 1-(hydrazino, Phenylhydrazino, Anilino and Methylamino) Phthalazine Derivatives (4b-e). A mixture of chlorophthalazine (4a) (0.01 mole) and each of hydrazine hydrate, phenylhydrazine, aniline and/or methylamine (0.01 mole) was refluxed in benzene (40 ml) for 6 hrs. The solids that separated on cooling were crystallized from the proper solvent.

Action of Acylhydrazines on Chlorophthalazine (4a): Formation of 1,2,4-triazolo-(4-triazolo-(4,3-b)-4,5-benzopyridazines (5a-c). A mixture of 1-chlorophthalazine (4a) (0.01 mole) and each of acylhydrazines, namely, benzoylhydrazine, phenylacetic hydrazide and/or salicyloylhydrazide (0.01 mole) in *n*-butanol (40 ml) was refluxed for 48 hrs. The solid separated after concentrating and cooling, recrystallized from the proper solvent.

Action of P_2S_5 on Phthalazinone Derivative (2a): Formation of Thione Derivative (2g). A mixture of (2a) (0.01 mole) and P_2S_5 (0.01 mole) in dry xylene (50 ml) was refluxed for 1 hr. The reaction mixture was filtered on hot. The solid obtained was recrystallized from the proper solvent.

Action of Copper Bronze on Thione (2g): Formation of the bis Compound (7). A mixture of the thione (2g) (0.01 mole) and copper bronze (1 gm) in dry xylene (40 ml) was refluxed for 10 hrs. The reaction mixture was filtered upon hot, the filtrate was concentrated. The solid separated was crystallized from the proper solvent.

Action of Grignard Reagents on Phthalazinone (2a): Formation of Phthalazine Derivative (8a and 8b). To an ethereal solution of methyl or ethylmagnesium iodide (0.02 mole), (0.01 mole) of phthalazinone (2a) in dry ether (50 ml) was added with stirring. The reaction mixture was refluxed on a steam bath for 6 hrs, left aside overnight at room temperature, then decomposed by saturated solution of aqueous ammonium chloride. The organic materials extracted with ether. The ethereal layer was dried over sodium sulphate anhydrous. Slow evaporation of ether solution, left solids which crystallized from the proper solvent.

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