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A Convenient Synthesis of 2-Arylindazol-3-Ones

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This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan, sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden. Terms & Conditions of access and use can be found at <u>http://www.tandfonline.com/page/terms-and-conditions</u> A CONVENIENT SYNTHESIS OF 2-ARYLINDAZOL-3-ONES

Clive W. Bird^{a*}, Joanne C. W. Chng^a, Nasim H. Rama^{b*} and Aamer Saeed^b.

- a) Department of Chemistry, King's College London, The Strand, London, WC2R 2LS, U.K.
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ABSTRACT: The reductive cyclisation of \underline{o} -nitrobenzanilides with zinc and sodium hydroxide in aqueous methanol provides a convenient source of 2-arylindazol-3-ones.

In connection with studies on the thermal rearrangement of 1-cyano-2-arylindazolones to benzimidazo-[2,1-b]quinazolones^{1,2} a convenient source of the precursor 2-arylindazolones was required. The most widely used routes to these compounds are the high pressure transition metal catalysed carbonylation of azobenzenes³, the cyclisation of <u>o</u>-arylhydrazinobenzoic acids⁴, the isomerisation of 3-aryl-2-hydroxyindazoles⁵, and the base catalysed cyclisation of <u>o</u>-azidobenzanilides⁶. All of these routes entail multiple steps from commercially available precursors and in some instances necessitate access to specialised equipment or the use of

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TABLE. Cyclisat	tion of	\underline{o} -Nitrobenzanilides	
Indazolone (II)	Yield	(%) m.p. (°C)	Ref.
Ar =			
Phenyl	45	208 - 210	1
<u>o</u> -Tolyl	39	229	6
<u>p</u> -Tolyl	27	212	6
<u>o</u> -Anisyl	30	72-74	10
<u>p</u> -Anisyl	46	170	6
3,4-Dimethylphenyl	30	198-200	10
<u>p</u> -Fluorophenyl	51	214-216	10
<u>o</u> -Chlorophenyl	30	183	6
<u>m</u> -Chlorophenyl	37	189 - 190	10
<u>p</u> -Chlorophenyl	30	230-233	6
<u>p</u> -Bromophenyl	34	220-222	10
<u>p</u> -Iodophenyl	11	196-198	10

potentially hazardous intermediates. An alternative solution to this problem was suggested by the solitary observation \overline{i} that 2-phenylindazolone was obtained from the reduction of <u>o</u>-nitrobenzanilide with zinc and sodium hydroxide.

We now wish to report that application of this approach to a variety of <u>o</u>-nitrobenzanilides (I) provides rapid and convenient access to the corresponding 2-arylindazolones (II) as exemplified by the examples assembled in the Table. The original reaction conditions⁷ have been modified so as to optimise the yield of 2-phenylindazolone and the yields quoted for other derivatives have not been corrected for the recovery of unreacted substrate as occurred in several instances.



The relatively modest yields of 2-arylindazolones obtained by this procedure are more than compensated for by the ready accessibility of <u>o</u>-nitrobenzanilides from commercially available precursors. Attempts to extend this method to the synthesis of 2-alkylindazolones have been thwarted by the greater ease of competing hydrolytic cleavage of N-alkyl-o-nitrobenzamides.

EXPERIMENTAL

General Procedure

A solution of the <u>o</u>-nitrobenzanilide^{8,9} (20 mmol) in methanol (55 ml) was added to sodium hydroxide(3.32 g, 83 mmol) in water (75 ml) together with zinc powder (3.92g, 60 mmol). The resulting reaction mixture was then heated under reflux with stirring for 15 hours and filtered hot to remove any remaining zinc. The filtrate was then concentrated to half the original volume and diluted with water. Any unreacted starting material was filtered off and the filtrate cooled in ice. This was then acidified with 1M aqueous sulphuric acid and the product extracted with ethyl acetate. The dried(Na₂SO₄) extracts were evaporated <u>in vacuo</u> and the residue crystallised from ethyl acetate-petroleum ether.

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