

SHORT COMMUNICATIONS

A PRELIMINARY REPORT ON THE EXCRETION OF ALUMINIUM IN RENAL FAILURE

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In recent years, the biological effects of aluminium have gained in importance. There are several evidences suggesting that the accumulation of aluminium causes fatal encephalopathy and a progressive osteomalacia in the last stages of patients with renal failure^{1,2}. Plasma and urine samples collected from a few healthy individuals and patients with renal failure were analysed by Zeeman corrected atomic absorptionometry. The results are summarized in table 1. The plasma values for aluminium were in the range 2–3 ppb, in normal healthy individuals, whereas in five patients of renal failure, the range was 500–825 ppb. Urinary excretion in normals was in the range 2–11 ppb whereas in the renal failure cases the range was 600–825 ppb. Patients suffering from kidney failure were given aluminium compounds along with antacids to accelerate phosphate depletion. How far such therapeutic regimens account for the higher levels of plasma and urinary aluminium remains to be studied.

Plasma and urine samples were used as such. 20 μ l aliquots were diluted with an equal volume of

Table 1 Aluminium excretion in normal individuals and patients with renal failure

	Patient examined	Aluminium (ppb)	
		Plasma	Urine
Healthy individuals	1	3	11
	2	2	7
	3	2	6
	4	3	8
	5	2	5
Individuals with renal failure	1	450	825
	2	525	600
	3	700	750
	4	650	725
	5	50	650

a solution containing 0.01 M HNO₃ and 0.1% Triton X. A Perkin Elmer (5000 AA) spectrometer with HGA 600 atomizer and AS-40 auto sampler were used at hollow cathode lamp current 25 mA with standard spectral line 396.2 nm. Samples were atomized in pyroelectrically coated graphite tubes.

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1. Arfrey, A. C., LeGendre, C. R. and Kachny, W. D., *N. Engl. J. Med.*, 1976, **294**, 184.
2. Jan Rud Anderson, *23rd Coll. Spectrosc. Internationale*, 1985, p. 444.

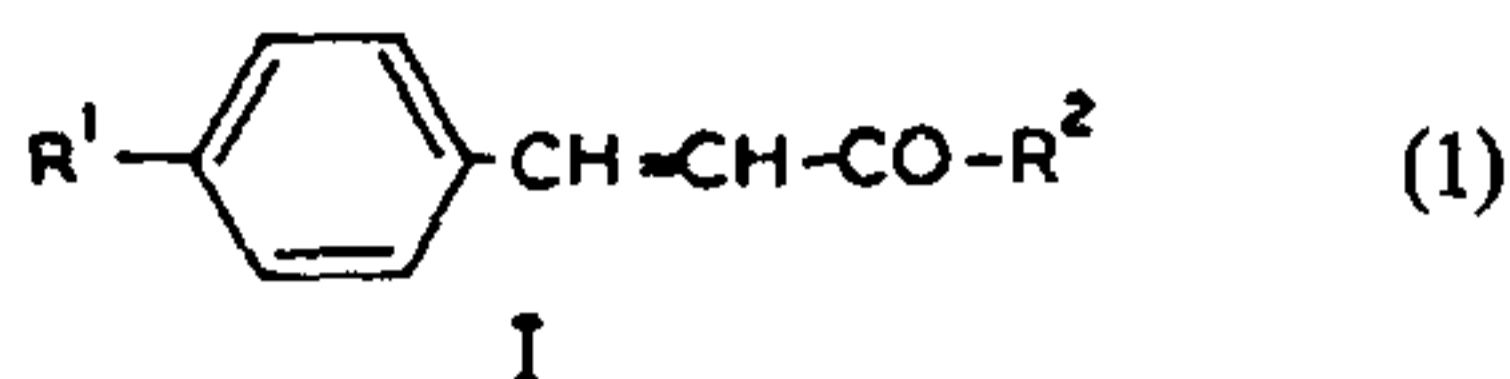
CORROBORATIVE STUDIES ON THE HIGHLY EFFICIENT PREPARATION OF 2'-HYDROXYCHALCONES USING PARTIALLY DEHYDRATED BARIUM HYDROXIDE CATALYST

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HYDROXY(METHOXY)SUBSTITUTED 2'-hydroxychalcones (II) occupy a central place in the biogenesis and in the synthesis of the various flavonoids¹. Claisen-Schmidt condensation between a 2-hydroxyacetophenone and an aromatic aldehyde in the presence of concentrated aqueous alkali (10–30%) is the oldest, simplest and most frequently used reaction for their synthesis. The reaction is generally carried out at room temperature for 24–28 h or at reflux (aq-ethanol) for 1–2 h. The various reaction conditions that have been used have been reviewed². Pinkey *et al*³ reported better results in the usual Claisen-Schmidt reaction in the presence of triethyl benzyl ammonium chloride (TEBA) under homogeneous conditions (NaOH-H₂O-ethanol-TEBA, 24 h at 30°); tetrabutyl ammonium iodide and tetrabutyl ammonium hydrogen sulphate proved less efficient. Recently Garcia-Raso^{4,5} and co-workers found that partially dehydrated commercial barium hydroxide efficiently catalyses Michael reaction of chalcones with active methylene compounds and Claisen-Schmidt condensation. The efficacy of this (heterogeneous) catalyst has been

shown by preparing styryl ketones of the type (1) where R^1 is H, OCH_3 , Cl, NO_2 or CH_3 and R^2 is an alkyl (17 examples) or a phenyl group (2 examples) and more recently Alcantara *et al*⁶ reported the preparation of 2'-hydroxychalcones containing different substituents in the B-ring only.



The desirable features of this condensation attracted our attention and we present below our results on the preparation of many other chalcones with oxygenation pattern typical of flavonoids. The results are summarized in table 1. Barium hydroxide octahydrate itself gave poorer results. A marginally lower yields are obtained in the chalcones (8-12). In all the cases the products obtained were of high purity.

We have not observed the formation of a flavanone arising out of the base catalysed cyclization of the 2'-hydroxychalcone and also Cannizzaro reaction products. This method has probably the same limitation as with other procedures in that it is not

applicable to 4-hydroxyacetophenones, 2 and 4-hydroxybenzaldehydes, though hydroxyl groups can be present in other positions.

In conclusion, the results of Alcantara *et al* and our own clearly establish that (partially) dehydrated barium hydroxide catalysed Claisen-Schmidt reaction is the most convenient, economical and practical method compared to all other procedures for rapid preparation of 2'-hydroxychalcones.

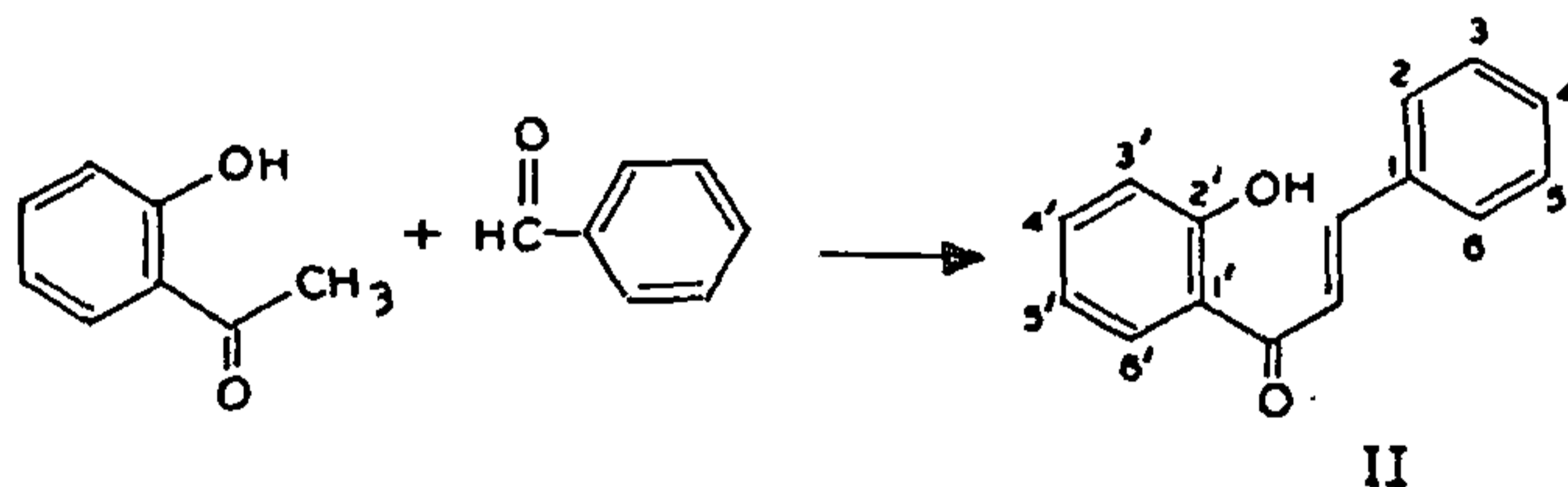
Preparation of barium hydroxide catalyst

Commercial $Ba(OH)_2 \cdot 8H_2O$ was heated in an oven at 200° for 2 h. The mass was powdered and stored in a desiccator. It is soluble in water and sparingly soluble in alcohol. A gravimetric determination of Ba^{2+} gave values corresponding to $Ba(OH)_2 \cdot 0.5$ to $1 H_2O$.

Preparation of 2'-hydroxy-4',6',3,4-tetramethoxychalcone

A mixture of 2-hydroxy-4,6-dimethoxyacetophenone (1.95 g, 10 mmol), Veratraldehyde (1.65 g, 10 mmol) and the barium hydroxide catalyst (500 g, excess) in 20 ml of ethanol (95%) was refluxed for

Table 1 Activated barium hydroxide catalysed preparation of 2'-hydroxychalcones



Sl. No.	Substituents in the chalcone					Yield ^a (time)	Obs.	M.p. (°C) Lit.
	3'	4'	6'	3	4			
1	H	H	H	H	H	90(1 h)	88	88 ⁶
2	H	OH	H	H	H	No reaction	—	—
3	H	H	H	H	OH	No reaction	—	—
4	H	H	H	OH	H	85(1 h)	168-69 ^b	—
5	H	H	H	H	OCH_3	89(1 h)	92-93	93 ⁷
6	H	OBz	H	H	OCH_3	85(1 h)	131-132	131-132 ⁸
7	H	OCH_3	H	H	H	86(1 h)	104-105	105 ⁹
8	H	OCH_3	OCH_3	H	H	85(2 h)	90-91	90-91 ¹⁰
9	H	OCH_3	OCH_3	H	OCH_3	80(2 h)	112-113	113 ¹¹
10	H	OCH_3	OCH_3	OCH_3	OCH_3	80(2 h)	156-157	157 ¹¹
11	H	OCH_3	OCH_3	OH	OCH_3	80(2 h)	196-197	196-197 ¹²
12	OCH_3	OCH_3	OCH_3	H	H	75(2 h)	140-141	141 ¹³

^aYields refer to isolated yields after one crystallization; ^bFound C, 74.3; H, 4.98%. Cal.: C, 75.00; H, 5%.

2h. The reaction mixture was poured into crushed ice and acidified with hydrochloric acid. The residue was isolated and crystallized from ethanol to yield 2'-hydroxy-4',6',3,4-tetramethoxychalcone (2.75 g, 80%).

The 2'-hydroxy chalcones prepared now have been identified by mixed m.pt with samples available from our sample collection, co-TLC and also by ^1H n.m.r.

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1. Harborne, J. B., Mabry, T. J. and Mabry, H. *The flavonoids*, (eds) Chapman and Hall, London, 1975.
2. Dhar, D. N. (ed.), *The chemistry of chalcones and related compounds*, Wiley Interscience, New York, 1981, p. 8.
3. Pinkey, Jain, P. K. and Grover, S. K., *Curr. Sci.*, 1983, **52**, 1185.
4. Garcia-Raso, A., Garcia-Raso, J., Campaner, B., Mestres, R. and Sinisterra, J. V., *Synthesis*, 1982, 1037.
5. Sinisterra, J. V., Garcia-Raso, A., Cabello, J. A. and Marinas, J. M., *Synthesis*, 1984, 502.
6. Alcantara, A. R., Marinas, J. M. and Sinisterra, J. V., *Tetrahedron lett.*, 1987, **14**, 1515.
7. Adams, R., *J. Am. Chem. Soc.*, 1919, **41**, 247.
8. Mahal, H. S., Rai, H. S. and Venkataraman, K., *J. Chem. Soc.*, 1935, 866.
9. Goel, R. N., Jain, A. C. and Seshadri, T. R., *Proc. Indian Acad. Sci.*, 1958, **A48**, 180.
10. Yuirokimura, J., *Pharm. Soc. Jpn.*, 1940, **60**, 151.
11. Kostanecki, St. V. and Tambor, J., *Ber.*, 1904, **37**, 792.
12. Hoerhammer, L., Farkas, L., Wagner, H. and Ostermayer, J., *Magy. Kem. Folyoirat*, 1964, **70**, 392; *Chem. Abstr.*, 1964, **61**, 14769c.
13. Rao, K. V. and Seshadri, T. R., *Proc. Indian Acad. Sci.*, 1948, **A27**, 385.

CARBARYL AND ENDOSULPHAN INDUCED ALTERATIONS IN THE INTESTINAL α -AMYLASE ACTIVITY OF *PHERETIMA POSTHUMA*

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OUR environment is getting polluted day by day due to human activities, urbanization and rapid industrialization. Almost all the agrochemicals which are used to kill the pests ultimately reach the soil and disturb the population of non-target organisms like earthworms^{1,2} which are beneficial to our farmers as well as the future source of pollution control. The soil surface becomes highly contaminated due to the fall out of insecticides and worms are exposed to high concentrations of chemicals when they come out on the surface at night in search of their food. The importance of earthworms in the ecosystem is very well-reviewed³. The soil fertility is closely associated with the population of earthworms in the soil besides other factors and the population of earthworms is directly related with the microclimatic conditions of the soil.

It is well-known that earthworms are omnivorous⁴. They digest not only the dead organic matter of the soil but also decomposed animals, living protozoa, rotifers and other minute organisms⁵. The digestive enzymes of only a few oligochaetes have been studied^{6,7}. The presence of amylase in the intestinal caecae of the segments 22-26 of *Pheretima*⁶ and in every part of the gut was detected in *Lumbricus terrestris* and *Eisenia foetida*⁷. The effect of phosphamidon on α -amylase of *Pheretima posthuma* has been worked out⁸. We have attempted here to determine the effect of carbaryl (1-naphthyl methylcarbamate, Sevin) and endosulphan (1,2,3,4,10,10-hexachloro-1,5,5a-6,9,9a-hexahydro-6,9-methano 2,4,3-benzodioxathiepin-3-oxide, Thiodan) on the α -amylase (1,4- α -D-Glucan glucanohydrolase, E.C. 3.2.1.1.) activity of *P. posthuma*.

The mature worms *P. posthuma* were treated with 2 mg/kg, 4 mg/kg, and 8 mg/kg of carbaryl and endosulphan by moist soil treatment. The worms were sampled at 0.5 h, 1 h, 2 h, 4 h, and 24 h intervals alongwith the normal worms, washed first in tapwater and finally with distilled water. Each worm was incisioned, intestine was taken out and washed properly in chilled double distilled water. A 10% homogenate of whole intestine was prepared in