

Letters to the Editor

A preparative synthesis of indole by dehydrogenation of 4,5,6,7-tetrahydroindole over catalysts with a low palladium content

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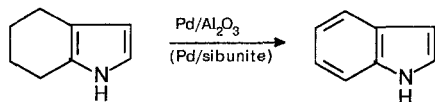
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Catalysts containing 0.15–0.5 % of Pd are highly active and selective in the dehydrogenation of 4,5,6,7-tetrahydroindole to indole when γ - Al_2O_3 or Sibunite are used as supports.

Key words: Pd-containing catalysts, tetrahydroindole, indole, dehydrogenation.

Synthetic and catalytic procedures for the preparation of indole from various organic compounds are known. Ref. 1 reports the results of dehydrogenation of the cyclohexene fragment into alkyl- and dialkyl-substituted indoles by boiling in xylene with 10 % Pd/C catalyst in a stationary system for 18 h.

In the present paper active and selective supported catalysts with low palladium content (0.15–0.5 %) for dehydrogenation of 4,5,6,7-tetrahydroindole (THI) and the corresponding reaction conditions for a flow system are found.



The results obtained are presented in Table 1.
The catalysts used were Pd/ γ - Al_2O_3 , PdS/ γ - Al_2O_3 ,

and Pd/Sibunite, the latter is pyrocarbon with a specific surface of 590 m^2/g , the corresponding catalyst contains 0.15–0.2 % of Pd. They were prepared by impregnation of the corresponding supports with PdCl_2 solution.

The experiments were carried out in a flow apparatus under H_2 . The catalyst (3–6 g) was placed into a quartz reactor (20×380 mm). A solution of THI in toluene (15–20 %) was supplied by means of a liquid pump. Indole was obtained in quantitative yield over Pd catalysts at 350–370 °C and a THI feed rate (ω) of 0.3–0.75 h^{-1} . The liquid products of the catalysis were fractionated *in vacuo*; indole was distilled at 146 °C/35 Torr, its m.p. 52.5 °C, b.p. 254 °C, and mass spectrum² are in agreement with those reported in the literature. The reaction products were analyzed by GLC (5 % OV-225 on Chromaton N 0.125–0.160 mm, 3×2000 mm column, the temperature program was 80 → 200 °C at 12 °C/min).

Thus, the results obtained show that active and se-

Table 1. Dehydrogenation of 4,5,6,7-tetrahydroindole on Pd catalysts ($p = 0.1$ mPa, toluene, $V_{H_2} = 3.6$ L h⁻¹)

Catalyst	THI content (%)	Exptl. conditions			Yield of cataly- sate (%)	GLC analysis (%)	
		$T/^\circ\text{C}$	ω/h^{-1}	τ/min		THI	Indole
0.25 % Pd/ γ -Al ₂ O ₃	20.8	370	0.75	30	100.0	—	100
0.15 % Pd/ γ -Al ₂ O ₃	18.3	365	0.60	25	100.0	—	100
(H ₂ S)							
0.2 % Pd/ γ -Al ₂ O ₃	15.5	350	0.52	74	93.5	—	100
(H ₂ S)							
0.15 % Pd/sibunitite	17.0	365	0.32	40	91.5	—	100
0.5 % Pd/sibunitite	18.2	364	0.70	35	95.9	Traces	100

lective Pd-containing supported catalysts containing small amounts of the metal and conditions were found for the preparative production of pure indole by dehydrogenation of THI in a flow system.

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References

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¹H, ¹³C NMR spectral data for α -, β -, and γ -lewisites and identification of *cis*, *trans*, *trans*- γ -lewisite as a new isomer

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The ¹H and ¹³C NMR spectral parameters of α -, β -, and γ -lewisites **1**–**5** were obtained and a new isomer, *cis,trans,trans*- γ -lewisite **5**, was isolated and identified on the basis of chemical shifts, relative intensities of the signals, and the intra-chain (³J_{HH}, ³J_{CH}) and inter-chain (³J_{CAsCH}) coupling constants.

Key words: lewisite; ¹H, ¹³C NMR spectra, new isomer *cis,trans,trans*- γ -lewisite.

A set of ¹H and ¹³C NMR parameters for *trans*- α -lewisite¹ and a partial set of ¹H and ¹³C{¹H} spectral data for *trans*- α -, β -, and γ -lewisites² have been described previously.

We have obtained a complete set of NMR parameters for all of the lewisite components (Table 1), synthesized as reported in Ref. 3: **1** + **2**, b.p. 55 °C (10 Torr); **3**, b.p. 90 °C (10 Torr); **4** + **5**, b.p. 130 °C (10 Torr).