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### DI-ISOPROPYLPHOSPHINODITHIOIC ACID

James L. Corbin<sup>a</sup>; William E. Newton<sup>a</sup>; John W. McDonald<sup>a</sup>

<sup>a</sup> C. F. Kettering Research Laboratory, Yellow Springs, Ohio

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## DI-ISOPROPYLPHOSPHINODITHIOIC ACID

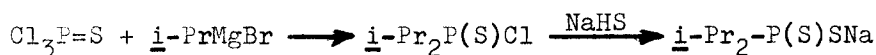
Submitted by  
(8/21/75)

James L. Corbin\*, William E. Newton  
and John W. McDonald

C. F. Kettering Research Laboratory#  
Yellow Springs, Ohio 45387

We have reported<sup>1</sup> some novel chemistry of molybdenum complexes containing the di-isopropylphosphinodithioate ligand, and now give the details of its synthesis by a route that we found to be most expeditious.

The title compound has been reported as a by-product from the reaction of readily available  $\text{PSCl}_3$  with  $\underline{i}\text{-PrMgBr}$ , the main product being  $\underline{i}\text{-Pr}_2\text{PSCl}$ . This suggested that the most direct route would be to treat the crude Grignard reaction



product with NaSH. Thus the entire synthesis would involve only two steps and avoid the isolation of an intermediate. This approach gave  $\underline{i}\text{-Pr}_2\text{PS}_2\text{H}$  in overall yields of 42-45%.

## EXPERIMENTAL

Di-isopropylphosphinodithioic Acid. - An argon blanket was maintained where feasible during the entire synthetic sequence, gloves were worn and all manipulations were performed in a fume hood. The Grignard reaction was run according to the literature procedure.<sup>2</sup>

## CFPI BRIEFS

The crude oil which was obtained after neutralization and the removal of solvent was dissolved in 150 ml of methanol and added to an NaSH solution (prepared by dissolving sodium (12.1 g) in methanol (150 ml), followed by saturation with H<sub>2</sub>S). The resulting solution was refluxed for 5 hrs, cooled, the sodium chloride was filtered, and the solvent was removed (rotary evaporator). The residue was dissolved in 200 ml of water, was washed with benzene (2 x 75 ml), was then cooled in ice and was acidified with conc. HCl (50 ml). The oily product was extracted into benzene (2 x 75 ml), was washed with water (2 x 50 ml) and was dried (Na<sub>2</sub>SO<sub>4</sub>). After removing the solvent (rotary evaporator), the residual oil was distilled (vacuum-jacketed Vigreux column) under vacuum<sup>3</sup> to give 38-41 g of colorless i-Pr<sub>2</sub>PS<sub>2</sub>H (92-94°/0.02 mm).

Nmr (CDCl<sub>3</sub>, 35°): The CH<sub>3</sub> protons resonated at 1.08, 1.19, 1.42, and 1.53 ppm, J<sub>PCCH</sub> = 20 Hz and J<sub>HCCH</sub> = 6.4 Hz. Other peaks were at 2.3 (m, CH) and 2.19 ppm (s, SH).

## REFERENCES

\*To whom correspondence should be addressed.

#Contribution No. 551.

1. (a) J. W. McDonald, J. L. Corbin and W. E. Newton, J. Am. Chem. Soc., 97, 1970 (1975); (b) "Proceedings of the International Symposium on Nitrogen Fixation," W. E. Newton and C. J. Nyman, Eds., Wash. State Univ. Press, 1975, in press.
2. P. J. Christen, L. M. van der Linde and F. N. Hooge, Rec. Trav. Chim. Pays-Bos., 78, 161 (1959).
3. The presence of some very low-boiling material, including H<sub>2</sub>S, required both liquid nitrogen and Dry-Ice/acetone traps to protect the pump.