MONTANA STATE UNIVERSITY

# The synthesis of 2-methyl butanal by reaction of n-butyl isocyanide with secondary-butylcadmium bromide, pt. 1 : a study of the reactions between carbon monoxide and the sodium salt of nitroethane, pt. 2

by John M Bruce Jr

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Montana State University
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Abstract:
Part I.

2-methyl butanal was prepared by the following sequence of reactions: [Formulas not Captured by OCR] A light yellow liquid was obtained as product. Confirmatory tests were run using small portions of the final product, and close agreement was found to exist between these results and the actual data pertaining to 2-methyl butanal. The 2,4-dinitrophenylhydrazone derivative was prepared from the final product, and its melting point determined.

Experimental details of the above procedure ere presented, along with an explanation of the experimental results.

Part II A reaction mixture consisting of the sodium salt of nitroethane and nickel carbonyl in a solvent of cyclohexane, was subjected to high pressures of carbon monoxide. This reaction mixture yielded, upon hydrolysis, a substance possessing a solubility which could not readily be accounted for. This substance is not obtained in the absence of nickel carbonyl, or when cyclohexane itself is subjected to carbon monoxide under pressure in the presence of nickel carbonyl. The unidentified substance is decomposed by steam distillation, simple distillation, and vacuum distillation, and cannot be crystallised satisfactorily even at low temperatures. Positive Schiff's and Tollen's tests were obtained by use of the appropriate reagent and the unknown substance. Derivatization of the unidentified material has not been possible.

Additional investigation of the unidentified substance is being conducted.

Part I. THE SYNTHESIS OF 2-METHYL BUTANAL BY REACTION OF n-BUTYL ISOCYANIDE WITH SECONDARY-BUTYLCADMIUM BROMIDE.

Part II. A STUDY OF THE REACTIONS BETWEEN CARBON MONOXIDE AND THE SODIUM SALT OF NITROETHANE.

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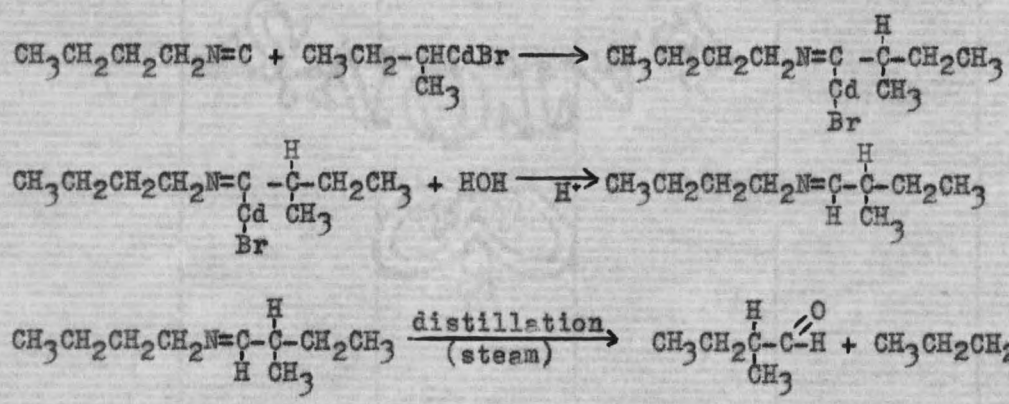
## I. ABSTRACT


### Part I.

2-methyl butanal was prepared by the following sequence of reactions:



A light yellow liquid was obtained as product. Confirmatory tests were run using small portions of the final product, and close agreement was found to exist between these results and the actual data pertaining to 2-methyl butanal. The 2,4-dinitrophenylhydrazone derivative was prepared from the final product, and its melting point determined.

Experimental details of the above procedure are presented, along with an explanation of the experimental results.

## II. INTRODUCTION

The isocyanides, or carbylamines, RNC, exist as colorless liquids, which are insoluble in water, and have a particularly disagreeable odor. The vapors are very toxic, capable of causing dizziness and vomiting if inhaled over a sufficient period of time.

The carbylamines are very reactive compounds. Some general reactions involving isocyanides and other molecules are listed by Nef[20], Gilman[8], and Cohen[6]. It has been demonstrated that the isocyanide group enters into an addition reaction with the Grignard reagent; it also enters into reactions with sulfur and chlorine at temperatures above 100°C, with the formation of RNCS and $RNC(Cl)_2$, respectively. Furthermore, it adds acid chlorides to give alkylimido chlorides, RN:C(Cl)COR.

Sachs and Loevy[23] give the complete procedure for the preparation of benzaldehyde by a reaction involving phenylmagnesium bromide and methyl isocyanide. No other aldehydes are reported to have been synthesized by this method. On the basis of the results obtained from attempted interaction between phenylmagnesium bromide and various alaphatic and aromatic isocyanides, Gilman and Heckert[9] concluded that only methyl isocyanide was of sufficient reactivity to combine with the Grignard reagent.

The desirability of developing the method of Sachs and Loevy into a good aldehyde synthesis is apparent, since aldehydes are the starting materials from which a large number of important compounds may be made.

The selection of the organocadmium compound as an alternative organometallic compound was prompted by the conclusions drawn from the experimental work of Gilman and Nelson[10], and de Bonneville[3]. These workers