

in the acid solution and is therefore separated from iron; no analytical data are available, however, for guidance in forming an opinion.

Mathews<sup>1</sup> has described as a means of separating iron from titanium, zirconium, and some of the rare earths, a modification of Rothe's method. The mixed, dried chlorides of the elements are heated with absolute ether and dry hydrochloric acid gas; the ferric chloride dissolves, leaving as a residue the chlorides of the other element or elements.

Although not at present very promising from the standpoint of speed, the three methods last described have interesting possibilities and deserve investigation; unfortunately, the time for a study of their efficiency has hitherto been lacking, but it is hoped that some experiments in this direction will shortly be carried out.

As a result of the study which has been made, it has been found that Baskerville's method is superior in accuracy to either of the others, but the writer is of the opinion that the modification of Gooch's method, described by Blair, is the best which has yet been made public. An excellent and detailed description of this process is given by Pope;<sup>2</sup> it does not take more time to carry out than does Baskerville's, and in the hands of the writer, has proved the more accurate.

FACULTY OF APPLIED SCIENCE,  
UNIVERSITY OF TORONTO, July, 1903.

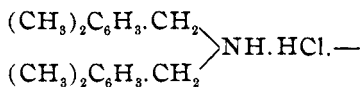
[CONTRIBUTION FROM THE LABORATORY OF THE UNIVERSITY OF MINNESOTA.]

## REDUCTION OF 2,5-DIMETHYLBENZALDAZINE AND THE PREPARATION OF SOME OF ITS SALTS.

BY EVERHART PERCY HARDING AND LILLIAN COHEN.

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*Preparation of the Hydrochloride of 2,5-Dimethyldibenzylamine.*<sup>3</sup>



—From 2 to 5 grams of 2,5-dimethylbenzalazine, prepared

<sup>1</sup> Mathews: This Journal, 20, 846.

<sup>2</sup> Pope: *Trans. Am. Inst. Min. Eng.*, 29, 372.

<sup>3</sup> The free base—dimethyldibenzylamine has not yet been analyzed.

according to the method of Curtius and Jay<sup>1</sup> from 2,5-dimethylbenzaldehyde, prepared by the Gatterman-Koch method,<sup>2</sup> were dissolved in 95 per cent. alcohol, some glacial acetic acid, and an excess of zinc dust added, and the flask with reflux condenser placed upon a water-bath, and the mixture gently boiled for about four hours. Upon the addition of very dilute sulphuric acid the unchanged azine was filtered off and to the filtrate, made strongly alkaline with sodium hydroxide, was added dilute hydrochloric acid. Upon standing some time, the hydrochloride crystallized out in the form of white, satin-like needles which crystallized from dilute alcohol in the form of colorless, prismatic needles and which melted at 227°. An analysis showed the following percentage composition:

	Calculated. Per cent.	Found. Per cent.
Carbon .....	74.74	74.70
Hydrogen.....	8.30	8.48
Nitrogen.....	4.84	4.91
Chlorine.....	12.11	12.10

2,5-Dimethyldibenzylaminehydrochloride is very soluble in ethyl and methyl alcohols and benzene, and slightly soluble in acetone. It is insoluble in cold but soluble in hot water.

*2,5-Dimethyldibenzylamine Nitrate*,  $C_{18}H_{22}N.HNO_3$ .—The nitrate was prepared by adding sodium hydroxide to a water solution of the hydrochloride and then treating the solution with dilute nitric acid. The nitrate precipitated at once as a white, crystalline substance which recrystallized out of dilute alcohol in the form of thin, colorless plates of the monoclinic system which melted at 215°. The analysis gave 8.86 per cent. N. Calculated, 8.80 per cent.

The nitrate is soluble in ethyl and methyl alcohols and insoluble in benzene. It is very difficultly soluble in acetone. It is soluble in hot, but insoluble in cold water.

*2,5-Dimethyldibenzylamine Picrate*,  $C_{18}H_{22}N.C_6H_2(NO_2)_3OH$ .—The picric acid derivative was prepared by adding to a concentrated alcoholic solution of the free base a concentrated alcoholic solution of the calculated amount of picric acid. The picrate was then precipitated by adding water. The voluminous precipitate was then filtered off, washed well with water and crystallized out of dilute alcohol. Upon crystallizing several times out of alcohol, the short sulphur-yellow prisms melted at 142°.

<sup>1</sup> *J. prakt. Chem.*, Neue Folge, 89, 43.

<sup>2</sup> *Ber. d. chem. Ges.*, 30, 1622.

The picrate is soluble in ethyl and methyl alcohols and very soluble in benzene and acetone. It is slightly soluble in hot but insoluble in cold water.

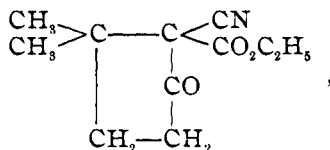
*2,5-Dimethyldibenzylamine Mercuric Chloride*,  
 $(C_{18}H_{23}N.HCl)_2.HgCl_2$ .—A concentrated water solution of mercuric chloride was added to a concentrated water solution of the hydrochloride. Upon standing, a white precipitate crystallized out which, upon recrystallizing out of alcohol, crystallized in long, colorless prisms which melted at  $157.5^\circ$ . The crystals are very soluble in ethyl and methyl alcohols and less soluble in benzene. They are very soluble in warm and slightly soluble in cold water.

*2,5-Dimethyldibenzylamine Chlorplatinate*,  
 $(C_{18}H_{23}HCl)_2.PtCl_4$ .—The platinum double salt was prepared by adding to a concentrated water solution of the hydrochloride a concentrated solution of chlorplatinic acid. The salt soon precipitated which recrystallized from alcohol in the form of reddish yellow, prismatic needles which melted at  $188^\circ$ . An analysis gave 21.22 per cent. of platinum, calculated 21.01 per cent. The double salt is soluble in ethyl and methyl alcohols and in acetone. It is insoluble in benzene and in hot and cold water.

## SYNTHESIS OF $\beta$ -METHYLADIPIC ACID.<sup>1</sup>

BY WILLIAM A. NOYES AND IRVING J. COX.

SOME time ago<sup>2</sup> one of us found that dimethylcyanocarboxethylcyclopentanone,



is decomposed by sodium hydroxide with formation of the sodium salts of malonic and hydroxyisocaproic acids. So far as we are aware, no other similar elimination of a carbon atom, by saponification, from a cyclic compound has been observed, and it seemed of interest to determine whether the reaction is a general one. To throw further light on this question, we have attempted to

<sup>1</sup> The work here described formed the basis of a thesis for the degree of Bachelor of Science at the Rose Polytechnic Institute.

<sup>2</sup> This Journal, 23, 396.