COMMUNICATIONS

DE LA FACULTÉ DES SCIENCES DE L'UNIVERSITÉ D'ANKARA

Série B : Chimie

TOME 15 B ANNEE 1968

Preparation of Hexamethylmelamine (2, 4, 6- Trisdimethylamino - 1, 3, 5- Triazine)

by

TURGUT GÜNDÜZ

Faculté des Sciences de l'Université d'Ankara
Ankara, Turquie

Communication de la Faculté des Sciences de l'Université d'Ankara

---

La Revue "Communications de la Faculté des Sciences de "Université d'Ankara" est un organe de publication englobant toutes les disciplines scientifiques représentées à la Faculté : Mathématiques pures et appliquées, Astronomie, Physique et Chimie théoriques, expérimentales et techniques, Géologie, Botanique et Zoologie.

La Revue, à l'exception des tomes I, II, III, comprend trois séries

Série A : Mathématiques, Physique et Astronomie.

Série B : Chimie.

Série C : Sciences naturelles.

En principe, la Revue est réservée aux mémoires originaux des membres de la Faculté. Elle accepte cependant, dans la mesure de la place disponible, les communications des auteurs étrangers. Les langues allemande, anglaise et française sont admises indifféremment. Les articles devront etre accompagnés d'un bref sommaire en langue turque.

Adrese: Fen Fakültesi Tebliğler Dergisi, Fen Fakültesi, Ankara, Turquie.

Preparation of Hexamethylmelamine (2, 4, 6- Trisdimethylamino - 1, 3, 5- Triazine)

TURGUT GÜNDÜZ

*(Faculty of Science University of Ankara, - Ankara, Turkey)**

Received 4. June. 1968

Triazines have proved of great interest, academic as well as industrial within the last two decades.

Since hoxamethylmelamine is starting material to the majority of the triazines, it holds a special place among these compounds. So the preparation of it with a high yield has been one of the main goals in triazine chemistry. While the latest method gives a 37 % yield, by the present work the yield in increased to as high as 93 %

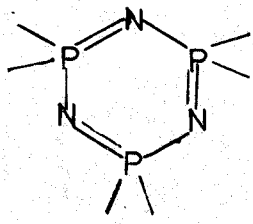
INTRODUCTION

A group of compounds containing hydrogen in place of carbon in the benzene ring in positions 1, 3, 5 are called triazines, well known heterocyclic systems. One of these triazines is hexamethylmelamine (2, 4, 6- trisdimethylamino- 1, 3, 5- triazine).

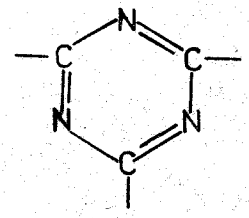
For a number of reasons, the triazines have proved of great interest, academic as well as industrial, within, the last two decades, (1) (2) (3) (4).

Though the the history of these compounds dates back to 1886 (5) (6) (7) the chemistry of them is not fully understood. The reason for this is probably the complicated resonance of triazine structure.

Triazines show great similarities to phosphazenes.



phosphazenes

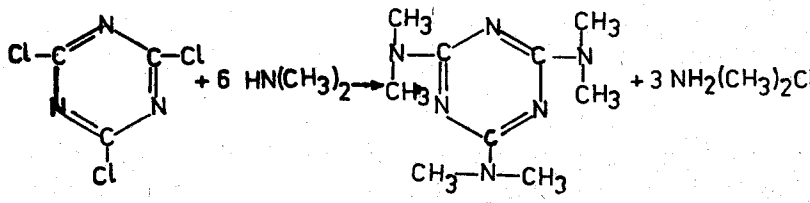


triazines

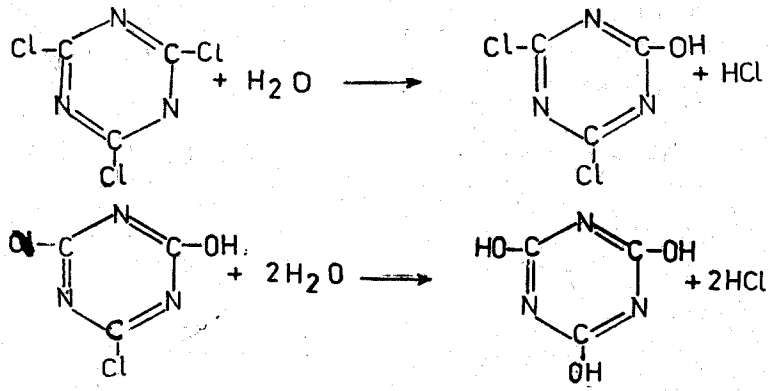
* Mailing address: Fen Fakültesi, Ankara Üniversitesi Ankara, Turkey

Since hexamethylmelamine is starting material for a number of triazines both scientific and industrial (1) the synthesis of it with a high yield offers a special interest. The latest method given to systhesize it produces only a 37 %yield (8). By the new method presented below it is possible to synthesize it with a high 93 % yield.

Three formulagrams of dimethylammonium chloride formed as side product, can easily be gained back by treating them with an aqueous sodium hydroxide solution.



Cyanuric chloride is a moisture - sensitive compound. It hydrolyses partly or completely even in the open air. So it is advisable to use it after recystallization from carbontetrachloride.



EXPERIMENTAL

74 gram (0, 4M) of cyanuric chloride, freshly crystallized from carbontetrachloride, dissolved in 400 ml of dry acetone (c. pure) is placed in a 1- litre three - necked round - bottomed flask equipped with a mercury sealed electric stirrer, dropping funnel and a special reflux condenser cooled with dry-ice mounted on an ordinary reflux condenser, Fig. 1.

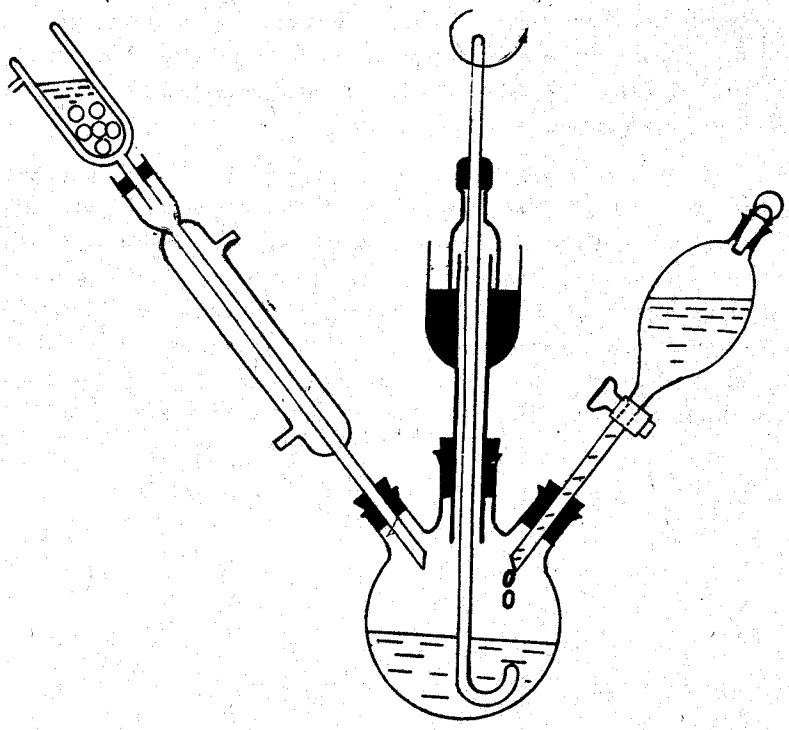


Fig. 1

150 ml (103 g. 2. 4M) dimethylamine, cooled by a mixture of dry - ice and acetone, is mixed with 150 ml of dry acetone cooled in the same manner. The solution is put in a dropping funnel and added dropwise within 3 hours into the solution of cyanuricchloride stirred vigorously by an electric stirrer. During the additon of the solution, especially at the beginning, the flask is cooled

externally by ice-water, and volatile components of the mixture are refluxed back by the condenser, cooled with acetone and dry-ice mixture. After some time, white crystals appear in the flask due partly to the formation of dimethylammonium chloride and partly to that of hexamethylmelamine (saturated solutian). When the addition of dimethylmelamine has ended the reaction mixture is stirred for three hours more, and then the mixture is left overnight. White precipitate is filtered off by a Buchner funnel and the filtrate evaporated until down to one fourth of its original volume. The slurry mixture, obtained by evaporation and the precipitate previously obtained are both added into 2 litres of distilled water, stirred carefully for some time and then filtered off. The crude product weighs 83.5 grams.

The crude substance is dissolved in 1 litre of dry methanol over a water bath then left to be crystallized which occurs first at room temperature and then in ice-cold water. After filtration of needlelike crystals, the filtrate is evaporated until it becomes saturated again and is left first at room temprature and then in ice-cold water. This procedure is repeated three times. The pure product so obtained weighs 78 gram corresponding to 93 % of the theoretical amount; it melts at 173 - 74C.

REFERENCES

[1] Smolin, E. M. and Rapaport L. The Chemistry of Heterocylic Compounds. Triazines and Derivatives. Interscience, New York 1959.

[2] Paddock, N. L. Quart. Rev. Chem. Soc. (1964) **18**, 169.

[3] Keat, R. and Shew, R. A. T. Chem. Soc. (1965). 4802.

[4] S. K. Das, R. A. Shaw, B. C. Smith, W. A. Last and F. B. G. Vells. Soc. Chem. Ind. (1963), 25

[5] H. N. Fries, Ber. (1886), **19**, 242, 2055.

[6] O. Diels, Ber, (1899), **32**, 691

[7] E. Freidheim J. Chem. Soc, (1944), **66**, 1775

[8] D. W. Kaiser, J. T. Thurston, J. R. Dubley, F. C. Schaefer. J. Hechenbleikner J. Am. Chem. Soc. (1951), **73**, 2984

ÖZET

Triazin bileşikleri geçen son yirmi yıl içinde hem akademik ve hem de endüstriel önem kazanmağa başladı. Bu maddelerden birisi olan hekzametilmelamin, diğer bazı azinler. için çıkış maddesi olduğundan, iyi bir verimle elde edilmesi triazin kimyası için mühim önemi haizdi. Bu noktadan hareket edilerek maddenin sentezi için yeni bir metod geliştirildi ve bu metod sayesinde % 37 olan verim % 93 çıkarıldı.

Prix de l'abonnement annul 1967:

Turquie : 15 TL ; Etranger : 30 TL.
Prix de ce numéro : 5 TL (paurla vente en Turquie).
Prière be s'adresser pour l'abonnement à : Fen Fakültesi Dekanlığı
Ankara Turquie.

Ankara Üniversitesi Basımevi - 1968