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Serial Number 18030

JLR-62-N, No. 3

E. I. duPont de Nemours & Company

Jackson Laboratory

February 12, 1941

Preparation of Intermediates for
Miscellaneous Products Research

N40783

JLR-62-N, No. 3 Serial Number 18030

E. I. duPont de Nemours & Company

Jackson Laboratory

New Products Division

G. E. Holbrook

Progress Report

February 12, 1941

# Preparation of Intermediates for Miscellaneous Products Research

Group Leader - C. W. Croco

Work Done By - J. R. Schaffer Work Started - 12-23-40

Reported By - J. R. Schaffer Completed - 1-14-41

J. M. TINKER W. S. CALCOTT

ASSISTANT DIRECTOR DIRECTOR

# Preparation of Intermediates for Miscellaneous Products Research (Project No. 2079)

J. R. Schaffer

JLR-62-N, No. 3 Serial Number 18030

Object of the Investigation:

To prepare compounds for evaluation as Nullapon B substitutes.

Period Covered by this Report:

December 23, 1940 to January 14, 1941.

Historical Background:

The compounds were requested by the Miscellaneous Products Division.

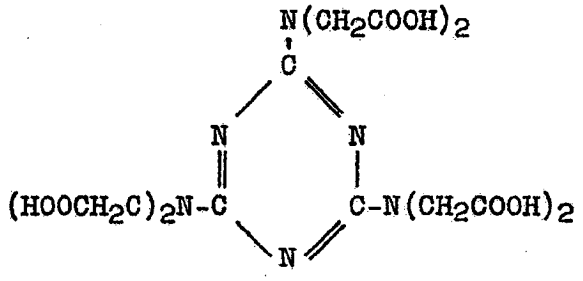
Summary and Conclusions:

N,N,N',N'-Tetracarboxymethyl-4-phenylenediamine, (208.2/1772)

was prepared by condensing 1 mol. of 4-phenylenediamine with 4 moles of chloroacetic acid.

The yield was 24.5% based on p-phenylenediamine.

Hexacarboxymethyl melamine, (309/3988)



was not obtained by reacting 1 mol. of melamine with 6 moles of chloroacetic acid. Melamine did not react with the chloroacetic acid under the conditions used.

Patent Situation:

The compounds are believed to be new but no action will be taken pending their evaluation.

## EXPERIMENTAL PART

N,N,N',N'-Tetracarboxymethyl-4-Phenylenediamine: (JLNB 3392-78)

A solution of the sodium salt of 4 moles of chloroacetic acid was added to a mixture of 1 mol. of p-phenylenediamine and 2 moles of soda ash in a 5-liter flask with stirring and the mixture was heated at 90-95°C. for 12 hours with stirring. One mol. additional of Na-salt of chloroacetic acid was added and the mixture was heated at 90-95°C. for 20 hours. The product was filtered off. Yield: 24.5% based on p-phenylenediamine.

Analysis: N = 6.98%,
Calculated for N,N,N',N'-tetracarboxymethyl-p-phenylenediamine-tetra-Na-salt = 6.54%.

Hexacarboxymethyl Melamine: (JLNB 3392-80)

A solution of the sodium salt of 6 moles of chloroacetic acid was added to a mixture of 1 mol. of melamine and 3 moles of soda ash in a 5-liter flask with stirring. The mixture was heated at 90-95°C. for 12 hours. When 1 mol. additional of the sodium salt of chloroacetic acid was added and heating at 90-95°C. was continued for 20 hours, the precipitate which separated out of the solution was filtered and dried at 50°C.

Analysis: N = 27.61%,
Calculated for hexacarboxymethyl melamine = N 17.72%,
Calculated for melamine, N = 66.7% which indicates that reaction did not take place.

Further work on this preparation was abandoned on advice of C. W. Croco.

Submitted for Typing
May 27, 1942

Typed June 8, 1942
RED