chloroform, carbon disulphide, acetic acid and in hot ethyl, methyl, and amyl alcohols. It is insoluble in water.

Nitroso and picric acid derivatives are at present being prepared.

PREPARATION OF 2,4,6-TRIMETHYLBENZALDAZINE; OF 2,4,6-TRIMETHYLBENZYL-2,4,6-TRIMETHYLBENZAL HYDRAZONE AND SOME OF ITS DERIVATIVES.

By Everhart Percy Harding.

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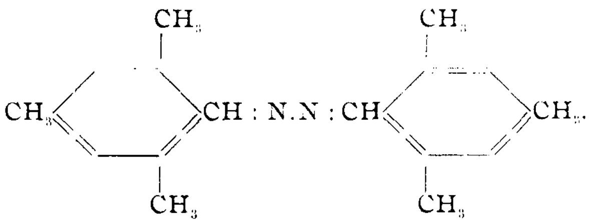
2,4,6-Trimethylbenzaldazine,



2,4,6-Trimethylbenzaldazine was prepared by adding to a weak alcoholic solution of 2,4,6-trimethylbenzaldehyde, which was prepared according to Gattermann,[1] a solution of hydrazine sulphate containing the calculated amount of sulphate to produce the corresponding aldazine. The mixed solutions were warmed to 60°, violently shaken for thirty minutes and allowed to stand for two hours. The yellow substance that separated formed spherulitic masses which recrystallized from acetic acid in beautiful light yellow prisms which melted at 167°.

$$(CH_3)_3C_6H_2CH\boxed{O + H_2}N'N\boxed{H_2 + O}HCC_6H_2(CH_3)_3 =$$
$$(CH_3)_3C_6H_2.CH : N.N : CH.C_6H_2(CH_3)_3.$$

These gave 9.59 per cent. N. Calculated, 9.56 per cent.

2,4,6-Trimethylbenzaldazine is very soluble in chloroform. It is soluble in cold ether, benzene, toluene, acetone and in hot acetic acid, ethyl, methyl and amyl alcohols. It is insoluble in water. Like all known aldazines it is a very stable compound.

2,4,6-Trimethylbenzyl-2,4,6-trimethylbenzal Hydrazone,

$$(CH_3)_3C_6H_2CH_2NHN : CHC_6H_2(CH_3)_3.$$

More than the calculated amount of a 4 per cent. sodium amalgam

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[1] W. Graf: Inaug. Diss., Heidelberg, 1899.

to reduce the aldazine to its corresponding hydrazone was added to an alcoholic solution of 2,4,6-trimethylbenzaldazine. This was heated at the temperature of boiling alcohol in a flask connected with a reflux condenser for from four to five hours upon a water-bath. The almost colorless solution was then filtered and the filtrate placed in ice water. The hydrazone crystallized at once. It was filtered off, washed well with water and recrystallized from alcohol. It melted at 88°-89° (uncorr.).

The analysis gave 9.40 per cent. N. Calculated, 9.53 per cent.

2,4,6-Trimethylbenzyl-2,4,6-trimethylbenzal hydrazone is very soluble in ether and benzene, less soluble in acetic acid, ethyl and methyl alcohols and insoluble in water. It is very unstable, decomposing at once when exposed to the air and after a short time in an exhausted desiccator.

Acetyl 2,4,6-Dimethylbenzyl-2,4,6-trimethylbenzal Hydrazone,

$(CH_3)_3C_6H_2CH_2N(C_2H_3O)N : CHC_6H_2(CH_3)_3$.

To a concentrated alcoholic solution of the hydrazone was added acetic anhydride in excess and the solution evaporated slowly to dryness upon a water-bath. The residue was recrystallized from alcohol. Long, satin-like, white needles were formed which melted at 155°.

The analysis gave 8.42 per cent. N. Calculated, 8.34 per cent.

Acetyl 2,4,6-trimethylbenzyl-2,4,6,trimethylbenzal hydrazone is very soluble in chloroform, ether, benzene, toluene, acetic acid and acetone. It is less soluble in ethyl, methyl and amyl alcohols and insoluble in water. It is a very stable compound.

Benzoyl 2,4,6-Trimethylbenzyl-2,4,6-trimethylbenzal Hydrazone,

$(CH_3)_3C_6H_2CH_2N(C_7H_5O)N : CHC_6H_2(CH_3)_3$.

A slight excess of benzoyl chloride was added to a concentrated alcoholic solution of the hydrazone and the solution evaporated to dryness upon a water-bath. The residue was taken up and recrystallized from alcohol. Long, satin-like needles were formed which melted at 142.5°-143°.

The analysis gave 7.03 per cent. N. Calculated, 7.23 per cent.

The benzoyl derivative is very soluble in chloroform, ether, toluene, acetone, benzene and acetic acid. It is less soluble in ethyl, methyl, and amyl alcohols, and is insoluble in water. It is a very stable compound.

Nitroso 2,4,6-Trimethylbenzyl-2,4,6-trimethylbenzal Hydrazone, $(CH_3)_3C_6H_2CH_2N(NO)N : CHC_6H_2(CH_3)_3$.

To a strong alcoholic solution of 2,4,6-trimethylbenzyl-2,4,6-trimethylbenzal hydrazone, cooled by ice water, was added a few drops of concentrated hydrochloric acid. To this cold solution, which was shaken after each addition, was added gradually a concentrated solution of sodium nitrite. Long, satin-like needles separated at once and increased in number upon the addition of water. The successful carrying-out of this experiment requires the use of freshly prepared hydrazone.

The nitroso derivative was filtered off, washed well with water to remove the excess of sodium nitrite, and recrystallized from alcohol. The long, yellow, satin-like crystals melted at 117°.

The analysis gave 13.09 per cent. N. Calculated, 13.00 per cent.

The nitroso derivative is very soluble in chloroform, ether, benzene, and toluene. It is less soluble in acetone and glacial acetic acid. It is soluble in warm ethyl, methyl, and amyl alcohols, and insoluble in water. It is a stable compound.

UNIVERSITY OF MINNESOTA.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF NORTH CAROLINA.]

ARSENIC PENTACHLORIDE.

BY CHARLES BASKERVILLE AND H. H. BENNETT.

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THE non-existence of arsenic pentachloride has for a long time been regarded as remarkable, especially so when the analogues of that element having atomic weights below and above it, phosphorus and antimony, show pentavalence toward the halogens.

Hurtzig and Geuther[1] endeavored to prepare it by treating arsenious acid with phosphorus pentachloride. Mayerhofer[2] passed chlorine into arsenic trichloride at —10° C., removing the excess of chlorine by a stream of carbon dioxide, and failed to obtain the body. Janovsky[3] treated phosphorus pentachloride with arsenic trihydride at 0° C. with negative results. Dumas[4]

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[1] *Ann. Chem. Pharm.*, **111**, 171.

[2] *Ibid.*, **158**, 326.

[3] *Ber. d. chem. Ges.*, **8**, 1636 (1875).

[4] *Ann. Chem.* (Liebig), **33**, 337.