of Triton B to form first N-cyanoethyl-N,N′-dibenzenesulfonyldiaminodurene, then a single N,N′ - dicyanoethyl - N,N′ - dibenzenesulfonyldiaminodurene. In one reaction when dicyanoethylation was accomplished in one step, *cis* and *trans* isomers were formed. The two N,N′-dicyanoethyl - N,N′ - dibenzenesulfonyldiaminodurenes hydrolyzed to the corresponding isomeric N,N - dicarboxyethyl - N,N - dibenzenesulfonyldiaminodurenes.

4. The lower-melting isomers of the N,N′-dialkyl - N,N′ - dibenzenesulfonyldiaminodurenes and of the N,N′-dicyanoethyl derivative were converted to the corresponding higher-melting isomers when heated above their melting points.

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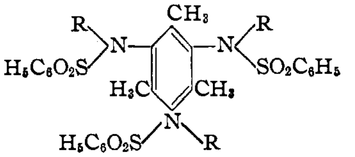
[CONTRIBUTION FROM THE NOYES CHEMICAL LABORATORY, UNIVERSITY OF ILLINOIS]

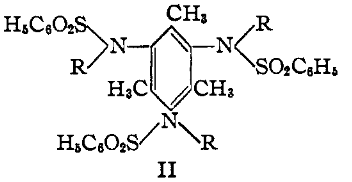
Restricted Rotation in Aryl Amines. X. Stereoisomers of N,N′N″-Trialkyl-N,N′,N″-tribenzenesulfonyltriaminomesitylenes

BY ROGER ADAMS* AND BRUCE ENGLUND[1]

Preceding papers in this series[2a,b] have reported the stereoisomerism of N,N′-dialkyl-N,N′-diarylsulfonyldiaminomesitylenes which arises from two points of restricted rotation in the molecule. The present paper describes the synthesis, separation and isomerization of pairs of isomeric N,N′,N″-trialkyl-N,N′,N″-tribenzenesulfonyltriaminomesitylenes (I and II). Although these molecules possess three points of restricted rotation, the other elements of symmetry present in them limit the number of possible stereoisomers to two, one *cis* and one *trans*.



I



II

R = CH_3, C_2H_5, $C_6H_5CH_2$, $H_5C_2O_2CCH_2$, HO_2CCH_2

These pairs of isomers were prepared following the general methods of synthesis of N,N′-dialkyl-N,N′-diarylsulfonyldiaminomesitylenes reported previously. Triaminomesitylene was obtained by catalytic reduction of trinitromesitylene. This was converted into the N,N′,N″-tribenzenesulfonyltriaminomesitylene by means of benzenesulfonyl chloride in pyridine at room temperature with a long reaction time. When N,N′,N″-tribenzenesulfonyltriaminomesitylene was treated in aqueous alkaline solutions with methyl iodide, ethyl iodide or benzyl chloride, two isomers of the corresponding N,N′,N″-trialkyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene were formed. Two isomers of N,N′,N″-tricarbethoxymethyl-N,N′,N″ - tribenzenesulfonyltriaminomesitylene were also obtained when ethyl bromoacetate was added to an anhydrous ethanol–acetone solution of N,N′,N″-tribenzenesulfonyltriaminomesitylene, made alkaline with sodium hydride. In this case a marked preponderance (9:1) of the less soluble, high-melting isomer was formed. Each of the isomeric esters gave a single acid upon hydrolysis and the two acids so obtained were isomeric.

The low-melting N,N′,N″-trimethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene isomer when melted, resolidified and melted again at the same point as the high-melting isomer with which it proved to be identical. Thermal isomerization of the *cis* form to the more stable *trans* form had apparently resulted. Parallel isomerizations were effected by fusing the low-melting forms of N,N′,-N″-triethyl- and N,N′,N″-tricarbethoxymethyl-N,N′,N″ - tribenzenesulfonyltriaminomesitylenes. The tribenzyl and tricarboxymethyl analogs decomposed under the conditions required for isomerization.

The infrared spectra of the pairs of isomers were determined and in each case served to establish the *cis–trans* isomerism. The spectra also established the purity of each of a pair of isomers with respect to the other.

In the benzenesulfonation of triaminomesitylene, difficulties were encountered. The procedure with benzenesulfonyl chloride and pyridine at 5° and short reaction time as followed for diaminomesitylenes gave very small amounts of mono- and di-benzenesulfonyl derivatives and a large amount of red oil. Under more vigorous conditions, the same products resulted with even larger quantities of undesirable by-products. Heating benzenesulfonyl chloride and triamino-

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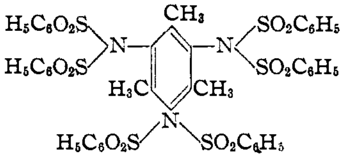
* Harvard University Ph.D. 1912.

[1] An abstract of a thesis submitted by Bruce Englund to the Graduate College of the University of Illinois, 1950, in partial fulfillment of the requirements for the degree of Doctor of Philosophy. He held the Standard Oil Company of Indiana Fellowship, 1948–1949.

[2] (a) Adams and Tjepkema, THIS JOURNAL, **70** 4204 (1948); (b) Adams and Rothstein, *ibid.*, **71**, 1620 (1949).

mesitylene without solvent resulted in extensive decomposition, but hexabenzenesulfonyltriaminomesitylene (III) was isolated from the mixture.



III

An additional previously unreported derivative of triaminomesitylene was prepared. In an attempt to synthesize N,N′,N″-triacetyltriaminomesitylene from acetic anhydride and triaminomesitylene trihydrochloride, Weidel and Wenzel[3] reported solvolytic loss of one amino group and characterized the product they obtained as 2,4-di-(acetamino)-6-acetoxymesitylene. Triacetaminomesitylene was readily prepared in an aqueous solution by the action of acetic anhydride and sodium acetate on triaminomesitylene.[4]

The authors are indebted to Mrs. J. L. Johnson and Miss Elizabeth Petersen for determination and interpretation of infrared spectra.

Experimental

Triaminomesitylene.—This material has previously been prepared[3,5] by reduction of trinitromesitylene with tin and hydrochloric acid. In this work the trinitro compound was reduced with hydrogen over a Raney nickel catalyst.

To a suspension of 124 g. of trinitromesitylene in 1000 ml. of absolute ethanol in a high-pressure bomb, 1.5 tablespoonfuls of Raney nickel was added and hydrogen introduced at an initial pressure of 1100 p. s. i. at room temperature. In ten minutes the temperature had risen to 65° and the theoretical amount of hydrogen had been absorbed.

The solution of triaminomesitylene was twice filtered through a Darco mat to remove catalyst and 500 ml. of solvent was distilled off. The remaining solution was allowed to cool and deposited a large crop of yellow crystals. Three additional crops were obtained from the mother liquor; total crude yield 66.2 g. (82%). After recrystallization from absolute ethanol 56.7 g. (71%) of product resulted, m. p. 123.5–125° (cor.). Weidel and Wenzel[3] reported 117–119°, Morgan[5] reported 118–119°; both used xylene for recrystallization.

N-Benzenesulfonyltriaminomesitylene.—To a stirred suspension of 16.5 g. of triaminomesitylene in 500 ml. of 10% aqueous sodium hydroxide were added two 40-ml. portions of benzenesulfonyl chloride at a fifteen-minute interval. The reaction mixture became warm and red-orange and after about fifteen minutes began to turn black. After forty-five minutes the reaction mixture was neutralized with hydrochloric acid, then made slightly acidic. The very sticky orange-red precipitate was filtered off and dissolved in 50 ml. of ethanol. The hot solution was diluted with water to the cloud point and deposited a very sticky brown precipitate when cool. This was boiled with 10 ml. of ethanol, leaving undissolved 0.4 g. (2%) of a product melting, after two recrystallizations from ethanol, at 224–225° (cor.). This melting point coincides with that of the dibenzenesulfonyl derivative, but the melting point of a mixture of the product with N,N′-dibenzenesulfonyltriaminomesitylene showed a depression of 20°.

Anal. Calcd. for $C_{15}H_{19}N_3O_2S$: C, 58.95; H, 6.27. Found: C, 59.07; H, 6.48.

N,N′-Dibenzenesulfonyltriaminomesitylene.—A suspension of 5.0 g. of triaminomesitylene in 35 ml. of pyridine was cooled to 0°. To the stirred suspension was added over a period of thirty minutes a solution of 16.2 ml. of benzenesulfonyl chloride in 50 ml. of pyridine. The temperature was not allowed to rise above 5°. After addition was complete, the ice-bath was removed and stirring continued for three hours. The reaction mixture was then poured into a mixture of 400 ml. of water, 250 g. of crushed ice and 50 ml. of concd. hydrochloric acid. A flocculent yellow precipitate appeared and was filtered off by suction after fifteen minutes. The mother liquor yielded a second similar crop.

The crude material was very impure and required manifold recrystallizations from ethanol to produce 1.7 g. (12.6%) of a product melting constantly at 224–224.5° (cor.).

Anal. Calcd. for $C_{21}H_{23}N_3O_4S_2$: C, 56.60; H, 5.16; N, 9.43. Found: C, 56.73; H, 5.31; N, 9.19.

The identity of this product was further demonstrated by its solubility in dilute hydrochloric acid and dilute sodium hydroxide solution and its diazotizability.

Hexabenzenesulfonyltriaminomesitylene.—To 1.25 g. of triaminomesitylene fused and heated in a bath at 140° was added dropwise 15 ml. of benzenesulfonyl chloride. The addition took forty-five minutes, after which 30 ml. of 25% aqueous sodium hydroxide was added to hydrolyze unreacted benzenesulfonyl chloride. The mixture, which had suffered considerable decomposition, was poured into 100 ml. of water. A very sticky red-brown precipitate was formed and separated by filtration. From this material only one product could be isolated in pure form. Extraction with 50 ml. of hot ethanol left a fine, slightly yellow powder undissolved which, after recrystallization from 20 ml. of 1:1 dioxane–water was pure white and weighed 0.2 g. (2.5%). It decomposed without melting at 330–335° (uncor.). The product was insoluble in aqueous alkali, which suggested that all hydrogen atoms on nitrogen had been replaced.

Anal. Calcd. for $C_{45}H_{39}N_3O_{12}S_6$: C, 53.75; H, 3.90; N, 4.19; S, 19.13. Found: C, 53.61; H, 4.09; N, 4.06; S, 19.24.

N,N′,N″-Tribenzenesulfonyltriaminomesitylene.—To a solution of 33 g. of triaminomesitylene in 450 ml. of pyridine was added in four equal portions at ten-minute intervals, a mixture of 155 ml. of benzenesulfonyl chloride and 150 ml. of pyridine. The stirred solution became very warm and turned from light yellow to a deep red-orange as the addition progressed. Stirring was continued at room temperature for thirty-five hours after which the reaction mixture was poured slowly with vigorous stirring into a mixture of 500 ml. of water, 500 g. of crushed ice, and 750 ml. of concd. hydrochloric acid. The very flocculent precipitate which formed immediately was stirred in the acid for forty-five minutes to ensure removal of pyridine.

The product was twice recrystallized from acetone–water; m. p. 243–244° (cor.). The yield was 70.7 g. (60.5%).

Anal. Calcd. for $C_{27}H_{27}N_3O_6S_3$: C, 55.35; H, 4.65; N, 7.18. Found: C, 55.31; H, 4.83; N, 7.40.

Isomers of N,N′,N″-Trimethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene.—To a solution of 3.3 g. of N,N′,N″-tribenzenesulfonyltriaminomesitylene, in 30 ml. of 20% aqueous sodium hydroxide and 20 ml. water, was added 1.1 ml. of methyl iodide dissolved in 40 ml. of ethanol. The resulting homogeneous solution was stirred at room temperature and precipitation of white crystals began after two hours. An additional 1.0 ml. of methyl iodide was introduced after sixteen hours. After twenty-two hours the product was filtered off. Concentration of the mother liquor produced two additional crops. The first crop weighed 1.6 g., m. p. 219–222° (cor.); the second crop weighed 0.2 g., m. p. 202–206° (cor.); the third crop weighed 0.4 g., m. p. 199–226° (cor.). The total

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(3) Weidel and Wenzel, *Monatsh.*, **19**, 250 (1898).

(4) Lumiere and Barbier, *Bull. soc. chim. France*, [3] **33**, 783 (1905).

(5) Morgan, *J. Chem. Soc.*, **123**, 228 (1923).

crude yield was 2.2 g. (62.2%), estimated to be about half and half of each isomer.

By extracting the mixture of three crops with 125 ml. of boiling ethanol and recrystallizing the undissolved portion from acetone–water, 0.9 g. of the pure less-soluble isomer resulted, m. p. 271.5–272.5° (cor.).

Anal. Calcd. for $C_{30}H_{33}N_3O_6S_3$: C, 57.46; H, 5.30. Found: C, 57.43; H, 5.09.

The ethanolic solution from the above extraction was evaporated to dryness and the product was crystallized from ethanol into four equal fractions. Fractions 3 and 4 were combined, dissolved and again separated into four fractions. Fractions 3 and 4 were again combined and the procedure repeated for a total of six times. Low-melting, more-soluble isomer was thus obtained pure, m. p. 208.5–209.5° (cor.).

Anal. Calcd. for $C_{30}H_{33}N_3O_6S_3$: C, 57.46; H, 5.30. Found: C, 57.46; H, 5.47.

Isomerization of N,N′,N″-Trimethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene (m. p. 208.5–209.5°).—In a centrifuge tube in a bath at 220°, 50 mg. of N,N′,N″-trimethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene (m. p. 208.5–209.5°) was fused and held in that condition for ten minutes. After cooling, the resulting material was powdered and a melting point of the product mixed with the high isomer (m. p. 271.5–272.5°) was determined and showed no depression. Before fusion, a melting point of the two isomers showed a depression of 9° below the melting point of the low-melting isomer.

A small portion of the low-melting isomer was dissolved in 10 ml. of glycerol and recovered unchanged by dilution with water. However, if the glycerol solution was heated to 220° for ten minutes, the only product recoverable melted at 270–271° (cor.) and was identical with the high isomer as shown by a melting point of the mixture.

Isomers of N,N′,N″-Triethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene.—To a solution of 11.7 g. of N,N′,N″-tribenzenesulfonyltriaminomesitylene in a mixture of 30 g. of potassium hydroxide in 200 ml. of water and 250 ml. of ethanol was added 10 ml. of ethyl iodide and the solution stirred at room temperature. Then, 10 ml. more of ethyl iodide was added after sixteen hours and again after forty hours. After stirring for fifty-two hours the reaction mixture was filtered. The white precipitate weighed 9.0 g., m. p. 208–237° (cor.). The mother liquor yielded a second crop (0.8 g., m. p. 205–232°) and a third crop (2.0 g., m. p. 159–175°). The total crude yield was 11.8 g. (91.8%).

The first and second crops were combined and extracted with 75 ml. of ethanol. The more-soluble isomer went into solution. The undissolved portion was recrystallized from acetone to which water was added to the cloud point, yielding 5.0 g. of the less-soluble isomer in pure form, m. p. 239–240° (cor.).

Anal. Calcd. for $C_{33}H_{39}N_3O_6S_3$: C, 59.17; H, 5.87. Found: C, 59.27; H, 6.04.

The ethanolic extract yielded a mixture of isomers. Isolation of the pure more-soluble form was more difficult than in the case of the methyl homolog. Repeated separation and combination of the more-soluble fractions, some from ethanol and some from acetone–water, eventually resulted in 650 mg. of pure product, m. p. 209–210.5° (cor.).

Anal. Calcd. for $C_{33}H_{39}N_3O_6S_3$: C, 59.17; H, 5.87. Found: C, 59.10; H, 5.91.

By the best estimate possible about 60% of the product formed was the high-melting isomer.

Isomerization of N,N′,N″-Triethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene (m. p. 209.5–210.5°).—After fusion of 50 mg. of N,N′,N″-triethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene (m. p. 209.5–210.5°) in a centrifuge tube held in a bath at 230° for fifteen minutes, and cooling, the product was recrystallized from acetone–water, m. p. 238–240° (cor.). The melting point of the mixture with the high-melting isomer showed no depression, whereas before fusion a mixture of high and low isomers showed a depression of 11° below the melting point of the low isomer.

Isomers of N,N′,N″-Tribenzyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene.—To a solution of 9.2 g. of N,N′,N″-tribenzenesulfonyltriaminomesitylene in a mixture of 200 ml. of 15% aqueous sodium hydroxide and 200 ml. of ethanol was added at room temperature with stirring 11.5 ml. of benzyl chloride. Small crystals began to appear in forty-five minutes. In sixteen hours, 6 ml. more of benzyl chloride was added. After stirring for nineteen hours, the white precipitate was filtered off, washed with water and ether and dried; weight 5.0 g., m. p. 235–238° (cor.). After addition of 11 ml. of benzyl chloride to the filtrate, stirring was continued for an additional seven hours, resulting in a second portion of product weighing 5.7 g., m. p. 235–238° (cor.). The filtrate was again stirred for four hours with 6 ml. of benzyl chloride producing 0.9 g. more of product, m. p. 235–238° (cor.). The large excess of benzyl chloride was required because of hydrolysis to benzyl alcohol, separation of which was noted after the first ten hours of stirring. The total crude yield was 11.6 g. (86%).

Separation of the two isomers was exceptionally difficult due to a very slight difference in solubility. The several crude crops were combined and extracted six times with 15 ml. each of acetone. The undissolved portion was recrystallized three times from methyl ethyl ketone. The pure product had an m. p. 251.5–253° (cor.). This product remained unchanged after being held at 260° in a fused condition for ten minutes.

Anal. Calcd. for $C_{48}H_{45}N_3O_6S_3$: C, 67.35; H, 5.29. Found: C, 67.51; H, 5.58.

The solid obtained from each of the acetone extracts after evaporation melted at 235–238°. The separate portions were recombined and boiled with 60 ml. of methyl ethyl ketone which was insufficient to effect complete solution. The undissolved portion was twice recrystallized from methyl ethyl ketone, giving a pure product, m. p. 273–274° (cor.).

Anal. Calcd. for $C_{48}H_{45}N_3O_6S_3$: C, 67.35; H, 5.29. Found: C, 67.57; H, 5.43.

Microscopic examination of the isomer, m. p. 251.5–253°, showed it to be long rhombohedral platelets in crystalline form, whereas the isomer, m. p. 273–274°, appeared as hard, tiny pyramids. Both forms could be seen in the fractions, m. p. 235–238°. Elementary analysis and the infrared spectrum of one of these latter fractions confirmed the conclusion that it was a mixture of the two isomers which were isolated in pure form.

Isomers of N,N′,N″-Tricarbethoxymethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene.—To a solution of 3.6 g. of sodium hydride in 250 ml. of anhydrous ethanol was added a solution of 29.25 g. of N,N′,N″-tribenzenesulfonyltriaminomesitylene in 250 ml. of anhydrous acetone. A solution of 25.0 g. of ethyl bromoacetate in 100 ml. of anhydrous ethanol was added and the mixture refluxed for three hours. Crystals began to appear in thirty minutes, but only a very slight amount appeared throughout the reaction time. The reaction mixture was placed in an ice box for two hours, during which time a large crop of white crystals formed. The precipitate was filtered by suction, washed with three small portions of ethanol, slurried with 200 ml. of warm water, filtered, and washed with water on the filter until free of bromide ion. The product weighed 26.5 g. (62.8%), m. p. 189–193° (cor.).

The mother liquor and ethanolic washings were combined and 350 ml. of solvent distilled off. The cooled solution deposited a precipitate which after washing and drying weighed 1.0 g., m. p. 170–182° (cor.). Further concentration to 100 ml. volume and dilution with an equal volume of water yielded a third crop which separated as an oil but crystallized overnight and which weighed 2.5 g., m. p. 134–140° (cor.). The total crude yield was 30.0 g. (71.2%).

The first and second crops were combined and extracted with 500 ml. of acetone. The undissolved portion and the first six of seven equal crops of crystals isolated from the acetone solution by evaporation were mixed and recrystallized from acetone. The yield was 21 g. of pure product, m. p. 195–195.5° (cor.).

Anal. Calcd. for $C_{39}H_{45}N_3O_{12}S_3$: C, 55.50; H, 5.37. Found: C, 55.67; H, 5.55.

The mother liquors from the above purifications yielded a by-product identified by analysis and infrared spectrum to be N,N′-dicarbethoxymethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene, m. p. 177.5–178° (cor.).

Anal. Calcd. for $C_{35}H_{39}N_3O_{10}S_3$: C, 55.47; H, 5.19; S, 12.69. Found: C, 55.52; H, 5.25; S, 12.44.

The third crop from the reaction mixture was recrystallized from ethanol seven times to yield a pure product, m. p. 148–148.5° (cor.).

Anal. Calcd. for $C_{39}H_{45}N_3O_{12}S_3$: C, 55.50; H, 5.37; S, 11.40. Found: C, 55.63; H, 5.36; S, 11.44.

It is noteworthy that the ratio of high-melting to low-melting isomer was approximately 9:1.

Isomerization of N,N′,N″-Tricarbethoxymethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene (m. p. 148–148.5°).—Upon fusion of 50 mg. of N,N′,N″-tricarbethoxymethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene (m. p. 148–148.5°) by immersion in a bath at 175–180° for forty-five minutes, the product, after cooling, was dissolved in 10 ml. of hot ethanol and the solution cooled, yielding a crop of white crystals, m. p. 193–194° (cor.). The melting point of a mixture of this product with the high-melting isomer showed no depression. A mixture of high and low-melting isomers before fusion melts gradually from 148–187° (cor.).

Isomers of N,N′,N″-Tricarboxymethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene.—To a solution of 8.44 g. of N,N′,N″-tricarbethoxymethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene (m. p. 195–195.5°) in 250 ml. of hot glacial acetic acid, 10 ml. of concd. sulfuric acid and 40 ml. water were added and the solution was refluxed gently for twenty hours. Solvent was evaporated with a stream of air to a volume of 100 ml. and 100 ml. of water was added. The white precipitate was filtered, washed with water and dried. It weighed 7 g. (92%). After two recrystallizations from 1:1 acetone–water, it melted at 260.5–261° (cor.) with decomposition.

Anal. Calcd. for $C_{33}H_{33}N_3O_{12}S_3$: C, 52.16; H, 4.38; N, 5.53; S, 12.66. Found: C, 52.04; H, 4.69; N, 5.32; S, 12.66.

A similar procedure was followed for the saponification of N,N′,N″-tricarbethoxymethyl-N,N′,N″-tribenzenesulfonyltriaminomesitylene (m. p. 148–148.5°). From 0.535 g. of ester, 0.445 g. (92%) of acid was obtained. After three crystallizations from a 2:1 benzene–acetone mixture the product melted at 235° (cor.) with decomposition. Three analyses of this product, even after vigorous treatment for removal of solvent, indicated that the acid had crystallized with one molecule of benzene.

Anal. Calcd. for $C_{33}H_{33}N_3O_{12}S_3 \cdot C_6H_6$: C, 55.90; H, 4.69. Found: C, 56.30; H, 4.87.

Attempts to obtain the acid free of benzene of crystallization by recrystallizing from aqueous acetone gave a product melting at 243–245° (cor.) with decomposition. Analysis indicated this to be the monohydrate.

Anal. Calcd. for $C_{33}H_{33}N_3O_{12}S_3 \cdot H_2O$: C, 50.95; H, 4.54. Found: C, 51.11; H, 4.63.

The compound containing benzene of crystallization was readily convertible to the monohydrate by boiling in water for two minutes, as shown by melting point and analysis. The reverse transition, confirmed by melting point, was also effected by boiling the monohydrate in dry benzene for five minutes and distilling off the solvent.

The two products were further differentiated by crystalline form. The acid crystallizes with benzene in long rhombohedral needles, whereas the monohydrate exists as very small irregular platelets.

Triacetaminomesitylene.—To a solution which had 3.3 g. of triaminomesitylene in 100 ml. of 0.6 *N* hydrochloric acid warmed to 50°, 8.0 ml. of acetic anhydride and then a solution of 6 g. of sodium acetate in 30 ml. of water were added with stirring. After two minutes a white precipitate began to appear. At the end of thirty minutes at 50°, the mixture was cooled and held at 10° for fifteen minutes, when the product was filtered by suction and dried. The mother liquor yielded two additional small crops of similar whiteness. The total crude yield was 2.9 g. (49.8%).

The product was surprisingly insoluble in the common organic solvents. It was purified by vacuum sublimation in a bath at 310° and at a pressure of 0.5 mm. The sublimate was pure white microcrystalline powder, m. p. 413–415° (uncor.), with decomposition.

Anal. Calcd. for $C_{15}H_{21}N_3O_3$: C, 61.83; H, 7.26. Found: C, 61.92; H, 7.42.

Summary

1. Triaminomesitylene formed by catalytic reduction of trinitromesitylene was converted to N,N′,N″-tribenzenesulfonyltriaminomesitylene by means of benzenesulfonyl chloride in pyridine. This derivative was then alkylated in alkaline solution with methyl iodide, ethyl iodide and benzyl chloride to give N,N′,N″-trialkyl-N,N′,N″-tribenzenesulfonyltriaminomesitylenes. A pair of isomers was obtained in each case.

2. In place of the alkyl groups, carbethoxymethyl groups were introduced in presence of sodium hydride. A pair of isomers resulted which after separation were hydrolyzed to the corresponding acids.

3. Three points of restricted rotation and symmetrical substitution in these molecules permit the formation of merely two isomers.

4. By heating benzenesulfonyl chloride and triaminomesitylene without solvent, extensive decomposition occurred but hexabenzenesulfonyltriaminomesitylene could be isolated from the reaction mixture. In aqueous solution, triaminomesitylene was acetylated with acetic anhydride to triacetaminomesitylene.

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