prismatic crystals melting at 179–180°. Admixture with that obtained before showed no depression of melting point.

Conversion of phenyl N,O-diacetyl-*p*-aminosalicylate (X) into phenyl *p*-aminosalicylate (I) by partial hydrolysis. 0.03 g. of phenyl N,O-diacetyl-*p*-aminosalicylate, dissolved in 0.3 g. of glacial acetic acid, was mixed with 0.3 ml. of 4 *N* hydrochloric acid and boiled for 5 minutes only and immediately cooled. About 5 ml. of water and then 1.2 ml. of 1 *N* sodium hydroxide were added. The precipitate which formed was collected and washed first with water, then with an excess of cold 2% sodium carbonate solution, and finally with water. To purify the crude product, it was dissolved in ice-cold 0.2 *N* sodium hydroxide solution, and the filtered, clear solution was saturated with carbon dioxide. The precipitated solid, after washing with water, was recrystallized from dilute methanol with decolorizing carbon and formed almost colorless, long prisms melting at 147°. It showed no depression of melting point on mixing with a sample of phenyl *p*-aminosalicylate, prepared from phenyl *p*-nitrosalicylate as described farther above, and in other respects is in accord with the latter.

Anal. Calcd. for $C_{13}H_{11}NO_3$ (229.09): C, 68.11; H, 4.84. Found: C, 67.83; H, 4.91.

We wish to thank heartily Director Dr. Satoru Kuwada for his kind encouragement through this work, and are also indebted to Misses H. Suzuki and Y. Kobayashi, Messrs. M. Kan, H. Kashiwagi and T. Nakata of our Analytical Section.

JUSO, HIGASHIYODOGAWA-KU
OSAKA, JAPAN
RECEIVED OCTOBER 30, 1951

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

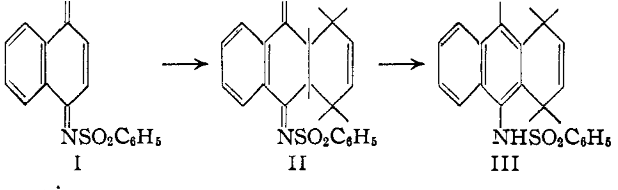
Quinone Imides. IX. Addition of Dienes to 1,4-Naphthoquinonedibenzenesulfonimide

BY ROGER ADAMS AND WILLIAM MOJE

1,4-Naphthoquinonedibenzenesulfonimide adds one molecule of butadiene, isoprene, dimethylbutadiene, chloroprene, cyclopentadiene, anthracene and α-acetoxybutadiene to give the corresponding 1,4,4a,9a-tetrahydro-9,10-anthraquinonedibenzenesulfonimides. The diimides formed from butadiene, isoprene, dimethylbutadiene and chloroprene are rearranged to 1,4-dihydro-9,10-anthracenedibenzenesulfonamides by treatment with mineral acid. The diimides from cyclopentadiene and anthracene dissociate when treated with mineral acid in acetic acid. The diimide from α-acetoxybutadiene when treated with mineral acid is converted to anthracene-9,10-dibenzenesulfonamide. This and 1,4-dihydro-9,10-anthracenedibenzenesulfonamide are oxidized with lead tetraacetate to anthraquinone-9,10-dibenzenesulfonimide which hydrolyzes to anthraquinone by the action of mineral acid. α-Acetoxybutadiene reacts with *p*-quinonedibenzenesulfonimide to give 5-acetoxy-4a,5,8,8a-tetrahydro-1,4-naphthoquinonedibenzenesulfonimide which is converted by mineral acid to 1,4-naphthalenedibenzenesulfonamide. This procedure is more convenient than any other previously described for the synthesis of 1,4-naphthalenedibenzenesulfonamide.

p-Quinonedibenzenesulfonimide has been shown to react with dienes[1] but in several instances the products did not correspond to those resulting from the action of dienes on *p*-benzoquinone. In the study of the addition of dienes to 1,4-naphthoquinonedibenzenesulfonimide milder conditions have been employed with excellent results.

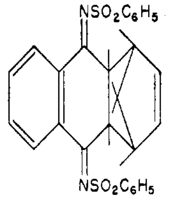
When 1,4-naphthoquinonedibenzenesulfonimide (I) in chloroform was treated with butadiene, isoprene or 2,3-dimethyl-1,3-butadiene, adducts separated. These were readily rearranged by warming with a little mineral acid. The products may be exemplified by the butadiene adduct 1,4,4a,9a-tetrahydro-9,10-anthraquinonedibenzenesulfonimide (II) which rearranged to 1,4-dihydro-9,10-anthracenedibenzenesulfonamide (III). This latter product (III) was also formed when the diimide (I) was heated at 100° with butadiene in benzene, the conditions used previously in the Diels–Alder additions to *p*-quinonedibenzenesulfonimide. Infrared spectra indicate the presence of the carbon–nitrogen double bond in type II and the NH group in type III.



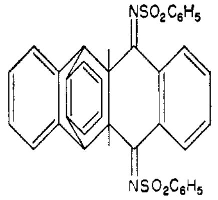
I II III

Chloroprene and 1,4-naphthoquinonedibenzenesulfonimide gave a product (probably of type II) which could not be crystallized but which was converted with hydrobromic acid in acetic acid to a well-defined product of type III.

Cyclopentadiene and anthracene added to 1,4-naphthoquinonedibenzenesulfonimide to give the diimides IV and V, respectively. The adducts are unstable and dissociate to the reactants on warming in organic solvents. Attempts to isomerize the adducts IV and V to the corresponding diamides by treatment with mineral acid in acetic acid were unsuccessful.



IV



V

The diamides of type III derived from butadiene, isoprene, dimethylbutadiene and chloroprene exhibit bright fluorescence when examined under ultraviolet light whereas the corresponding diimides and those from cyclopentadiene and anthracene do not. This fluorescence serves as a satisfactory method for the identification of the primary adducts from 1,4-naphthoquinonedibenzenesulfonimide.

α-Acetoxybutadiene has been added to *p*-benzoquinone and 1,4-naphthoquinone.[2] The adduct

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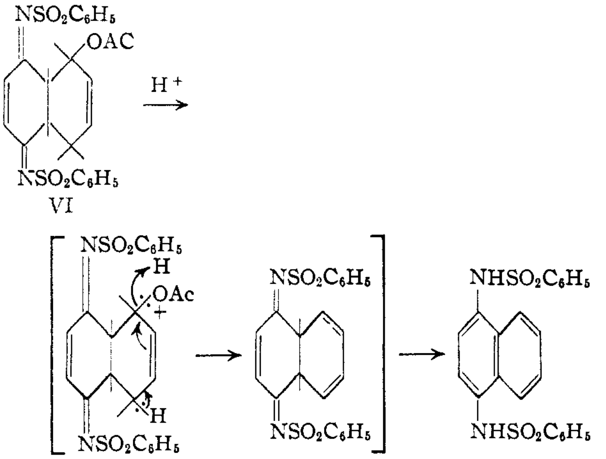
[1] R. Adams and C. R. Walter, THIS JOURNAL, **73**, 1152 (1951).

[2] H. Vollman, F. Schoffer and W. Ostrowski, I. G. Farb. A.-G. (German Patent 739,438); *Chem. Zentr.*, **115**, I, 184 (1944).

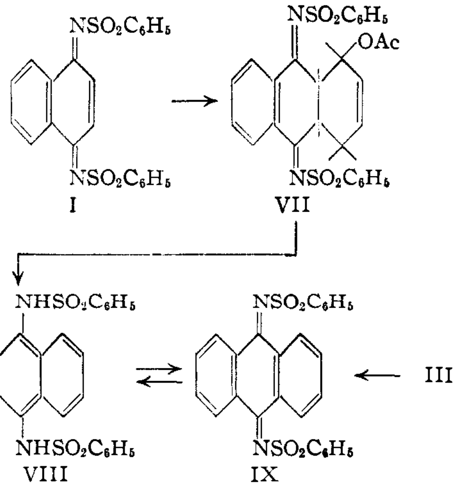
from 1,4-naphthoquinone was 1-acetoxy-1,4,4a,9a-tetrahydro-9,10-anthraquinone. *p*-Quinonedibenzenesulfonimide added α-acetoxybutadiene in a similar manner to give 5-acetoxy-4a,5,8,8a-tetrahydro-1,4-naphthoquinonedibenzenesulfonimide (VI) which rearranged with loss of acetic acid when treated with a little mineral acid in acetic acid. The product was 1,4-naphthalenedibenzenesulfonamide. The mechanism is probably



VI

This method was used for the preparation of large quantities of 1,4-naphthalenedibenzenesulfonamide in place of that involving the reduction of 4-nitro-1-naphthylamine to 1,4-naphthalenediamine and benzenesulfonation.[3]

Treatment of 1,4-naphthoquinonedibenzenesulfonimide in chloroform with α-acetoxybutadiene gave 1-acetoxy-1,4,4a,9a-tetrahydro-9,10-anthraquinonedibenzenesulfonimide (VII) which on treatment with hydrochloric acid in acetic acid was converted to 9,10-anthracenedibenzenesulfonamide (VIII). The adduct (VII) loses acetic acid much more readily than VI and the elimination can be achieved simply by heating.



I VII III VIII IX

9,10-Anthracenedibenzenesulfonamide (VIII) shows a bright yellowish-green fluorescence in ultraviolet light. Treatment of VIII with ferric chloride in acetic acid gave anthraquinone in 95% yield. Oxidation of VIII with lead tetraacetate or sodium bismuthate in acetic acid gave anthraquinonedibenzenesulfonimide (IX). A tetrahydrofuran solution of IX was reduced to VIII by treatment with aqueous sodium hydrosulfite. Treatment of IX in acetic acid with a few drops of hydrochloric acid gave anthraquinone almost quantitatively.

When III was treated with sodium bismuthate in acetic acid oxidation, dehydrogenation and hydrolysis took place and anthraquinone was formed. However, when III was oxidized with lead tetraacetate in acetic acid, anthraquinonedibenzenesulfonimide separated. This was identical with the product from the oxidation of VIII as shown by melting point and a comparison of infrared spectra. The lead tetraacetate served not only as an oxidizing agent but also as a dehydrogenating agent.

Acknowledgment.—The authors are indebted to Miss Emily Davis, Miss Jean Fortney and Mrs. Katherine Pih for the microanalyses and to Miss Elizabeth Petersen for infrared spectra.

Experimental[4]

Infrared spectra of the diimides, type II, have been found in this Laboratory to exhibit two medium to strong absorption bands at 1600 to 1620 cm.$^{-1}$ and 1560 to 1590 cm.$^{-1}$ and no absorption in the 3300 cm.$^{-1}$ region characteristic of the –NH– stretching frequency.[5] The amides, type III, have sharp medium absorption bands in a narrow region from 3275 to 3310 cm.$^{-1}$.

1,4,4a,9a-Tetrahydro-9,10-anthraquinonedibenzenesulfonimide.—A mixture of 10 g. of 1,4-naphthoquinonedibenzenesulfonimide, 15 g. of butadiene and 100 ml. of chloroform was allowed to stand at room temperature in a pressure bottle. After standing for five days the diimide had gone into solution and white needles of the adduct had formed. The crystals were removed by filtration giving 4 g. of slightly yellow needles. The chloroform filtrate was evaporated to 50 ml. and cyclohexane added giving 8 g. of very light pink needles. The combined products were recrystallized from chloroform–cyclohexane to give colorless needles weighing 11 g. (98%), m.p. 258–258.5° (dec.). The infrared spectrum showed the presence of the carbon–nitrogen double bond (1605, 1573, 1560 cm.$^{-1}$) and no -NH- grouping.

Anal. Calcd. for $C_{26}H_{22}N_2O_4S_2$: C, 63.65; H, 4.52; N, 5.71. Found: C, 63.85; H, 4.69; N, 5.74.

1,4-Dihydro-9,10-anthracenedibenzenesulfonamide.—A mixture of 2 g. of 1,4-naphthoquinonedibenzenesulfonimide and 3 g. of butadiene suspended in 15 ml. of benzene was heated in a sealed tube at 100° for 15 hours. The solid was collected and after washing with hot benzene and drying weighed 2 g. (89%). Recrystallization from glacial acetic acid gave colorless needles, m.p. 258–259° (dec.). The infrared spectrum showed the presence of the –NH– linkage (3295 cm.$^{-1}$) and no carbon–nitrogen double bond.

Anal. Calcd. for $C_{26}H_{22}N_2O_4S_2$: C, 63.65; H, 4.52; N, 5.71. Found: C, 63.46; H, 4.64; N, 5.75.

To a solution of 4.42 g. of 1,4,4a,9a-tetrahydro-9,10-anthraquinonedibenzenesulfonimide in 60 ml. of hot glacial acetic acid was added 3 drops of 48% hydrobromic acid. After cooling the white needles were collected and washed with petroleum ether (b.p. 30–40°). The yield was 4.08 g. (93%); m.p. 258–259° (dec.). The infrared spectrum of this compound and the compound described above were identical.

1,4,4a,9a-Tetrahydro-2-methyl-9,10-anthraquinonedibenzenesulfonimide.—To a solution of 10 g. of 1,4-naphthoquinonedibenzenesulfonimide in 100 ml. of chloroform was added 20 ml. of freshly distilled isoprene. After standing for four days the diimide that had separated on the addition of the isoprene had disappeared and the solution was a very light yellow in color. The solution was evaporated on the steam-bath to 75 ml. and 250 ml. of petroleum ether (b.p.

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(3) R. Adams and R. A. Wankel, THIS JOURNAL, **73**, 131 (1951).

(4) All melting points are corrected.

(5) H. M. Randall, R. G. Fowler, N. Fuson and J. R. Dangl, "Infrared Determination of Organic Structures," D. Van Nostrand Co., Inc., New York, N. Y., 1949.

30–40°) was added. The adduct crystallized in large colorless needles and weighed 11.3 g. (98%). The product was purified by recrystallization from glacial acetic acid. It melted at 189–192°, resolidified to a yellow solid which darkened at 233° and decomposed at 248°. The infrared spectrum showed the presence of the carbon–nitrogen double bond (1605–1612, 1581 cm.$^{-1}$) and no -NH- linkage.

Anal. Calcd. for $C_{27}H_{24}N_2O_4S_2$: C, 64.26; H, 4.79; N, 5.55. Found: C, 64.27; H, 5.01; N, 5.68.

1,4-Dihydro-2-methyl-9,10-anthracenedibenzenesulfonamide.—To a solution of 9.0 g. of the compound just described in 150 ml. of boiling glacial acetic acid was added 3 drops of 48% hydrobromic acid. After cooling the white needles that had separated were collected and washed with acetic acid. The yield was 8.1 g. (90%); m.p. 247–248° (dec.). The infrared spectrum showed the presence of the -NH- linkage (3291 cm.$^{-1}$) and no carbon–nitrogen double bond.

Anal. Calcd. for $C_{27}H_{24}N_2O_4S_2$: C, 64.26; H, 4.79; N, 5.55. Found: C, 64.15; H, 5.04; N, 5.75.

1,4,4a,9a-Tetrahydro-2,3-dimethyl-9,10-anthraquinonedibenzenesulfonimide.—To a solution of 4 g. of 1,4-naphthoquinone dibenzenesulfonimide in 40 ml. of chloroform was added 10 ml. freshly distilled dimethylbutadiene. After the solution had stood for 18 hours it was evaporated to 20 ml. and 100 ml. of hot cyclohexane was added. After cooling the colorless crystals that had separated were collected and washed with cyclohexane. The yield was 4.6 g. (97%). Recrystallization from glacial acetic acid gave small white needles, m.p. 233–234° (dec.), with softening at 193–198°. The infrared spectrum showed the presence of the carbon–nitrogen double bond (1607, 1582 cm.$^{-1}$) and no -NH- grouping.

Anal. Calcd. for $C_{28}H_{26}N_2O_4S_2$: C, 64.84; H, 5.05; N, 5.40. Found: C, 64.70; H, 5.26; N, 5.37.

1,4-Dihydro-2,3-dimethyl-9,10-anthracenedibenzenesulfonamide.—To a solution of 1 g. of the diimide just described dissolved in 50 ml. of hot glacial acetic acid was added 2 drops of 48% hydrobromic acid. After cooling the crystals that had separated were collected and washed with acetic acid. The yield was 0.85 g. (85%). Recrystallization from glacial acetic acid gave small white feathery needles, m.p. 233–234° (dec.). The infrared spectrum showed the presence of the -NH- linkage (3285 cm.$^{-1}$) and no carbon–nitrogen double bond.

Anal. Calcd. for $C_{28}H_{26}N_2O_4S_2$: C, 64.84; H, 5.05; N, 5.40. Found: C, 64.51; H, 5.33; N, 5.43.

1,4-Dihydro-2-chloro-9,10-anthracenedibenzenesulfonamide.—To a solution of 2 g. of 1,4-naphthoquinonedibenzenesulfonimide in 25 ml. of chloroform was added 10 ml. of freshly distilled chloroprene. After standing for 15 days colorless crystals had separated and the chloroform solution was pale yellow in color. Addition of petroleum ether (b.p. 30–40°) gave a cream colored precipitate which weighed 2.2 g. (91%). Since attempts to crystallize this product from various solvents were unsuccessful it was converted directly to the amide. To a solution of the 2.2 g. in 50 ml. of hot acetic acid was added 2 drops of 48% hydrobromic acid and then 100 ml. of hot cyclohexane. The crystals that separated were removed, dried and dissolved in 50 ml. of 1 *N* aqueous sodium hydroxide (Darco). Filtration and acidification of the filtrate gave a white precipitate, which weighed 1.1 g. Recrystallization from glacial acetic acid gave small white needles, m.p. 250.5–251° (dec.). The infrared spectrum showed the presence of the -NH- linkage (3291 cm.$^{-1}$) and no carbon–nitrogen double bond.

Anal. Calcd. for $C_{26}H_{21}ClN_2O_4S_2$: C, 59.48; H, 4.03; N, 5.34. Found: C, 59.44; H, 3.86; N, 5.25.

Adduct of Cyclopentadiene and 1,4-Naphthoquinonedibenzenesulfonimide.—To a solution of 3 g. of 1,4-naphthoquinonedibenzenesulfonimide in 60 ml. of hot glacial acetic acid was added 5 ml. of freshly distilled cyclopentadiene. When the light yellow acetic acid solution had cooled to room temperature, 3 ml. more of cyclopentadiene was added and the solution allowed to stand overnight. The colorless needles that separated were collected, washed with petroleum ether (b.p. 30–40°), and dried. The yield was 2.26 g. (66%). Heating dissociates the adduct. It gradually turns from yellow to brown (183°), softens at 190° and decomposes at 191–192°. Recrystallization from various solvents also causes dissociation of the adduct. The infrared spectrum showed the presence of the carbon–nitrogen double bond (1603, 1562 cm.$^{-1}$) and no -NH- linkage.

Anal. Calcd. for $C_{27}H_{22}N_2O_4S_2$: C, 64.52; H, 4.41; N, 5.58. Found: C, 64.32, 64.87; H, 4.66, 4.54; N, 5.40.

Adduct of Anthracene and 1,4-Naphthoquinonedibenzenesulfonimide.—A mixture of 2.18 g. of 1,4-naphthoquinonedibenzenesulfonimide and 0.89 g. of purified anthracene[6] in 50 ml. of benzene was heated at the boiling point for several minutes until the diimide dissolved. The dark green solution was allowed to stand at room temperature for 5 weeks. The stopper was then removed from the flask and the benzene allowed to evaporate at room temperature for one week. Small colorless needles separated. They were washed with 2 ml. of benzene and dried; weight 1.30 g. (42%). Recrystallization from acetonitrile gave pale yellow needles, m.p. 172–173° (dec.), with previous melting at 163° and resolidifying at 165°. The infrared spectrum showed the presence of the carbon–nitrogen double bond (1604, 1567 cm.$^{-1}$) and no -NH- grouping.

Anal. Calcd. for $C_{36}H_{26}N_2O_4S_2$: C, 70.34; H, 4.26; N, 4.56. Found: C, 70.33; H, 4.48; N, 4.60.

5-Acetoxy-4a,5,8,8a-tetrahydro-1,4-naphthoquinonedibenzenesulfonimide.—To a solution of 64 g. of *p*-quinonedibenzenesulfonimide in 600 ml. of hot chloroform was added 20 g. of α-acetoxybutadiene.[7] The solution was heated to the boiling point and allowed to stand at room temperature for 4 days. The colorless crystals weighed 41.5 g. Evaporation of the chloroform filtrate to 300 ml. gave an additional 6 g. The yield was 47.5 g. (58%). Recrystallization from glacial acetic acid gave small white rods, m.p. 214.5–215° with the evolution of a gas. The melt resolidified and remelted at 257–260° (dec.). The infrared spectrum showed the presence of the carbon–nitrogen double bond (1618, 1588 cm.$^{-1}$) and the carbon–oxygen double bond (1730 cm.$^{-1}$).

Anal. Calcd. for $C_{24}H_{22}N_2O_6S_2$: C, 57.81; H, 4.45; N, 5.62. Found: C, 57.58; H, 4.43; N, 5.42.

1,4-Naphthalenedibenzenesulfonamide.—To a solution of 40 g. of the above crude adduct in 700 ml. of boiling glacial acetic acid was added 10 ml. of concentrated hydrochloric acid. After cooling, the small colorless needles that had separated were collected on a filter and washed with glacial acetic acid and dried, m.p. 258–259° (dec.). The yield was 34 g. (97%). There was no depression when a melting point was taken of this compound with 1,4-naphthalenedibenzenesulfonamide.[3]

1-Acetoxy-1,4,4a,9a-tetrahydro-9,10-anthraquinonedibenzenesulfonimide.—To a solution of 8.73 g. of 1,4-naphthoquinonedibenzenesulfonimide in 80 ml. of chloroform was added 8 g. of 1-acetoxybutadiene. After standing at room temperature for 4 days the pale yellow solution was heated to the boiling point and 400 ml. of hot cyclohexane was added. The colorless crystals that separated were washed with petroleum ether (b.p. 30–40°). The yield was 9.79 g. (89%). Recrystallization from benzene–cyclohexane gave small white flat rods which melted at 242–243° (dec.) with yellowing at 203–213°. The infrared spectrum showed the presence of the carbon–nitrogen double bond (1609, 1573 cm.$^{-1}$) and the carbon–oxygen double bond (1736 cm.$^{-1}$).

Anal. Calcd. for $C_{28}H_{24}N_2O_6S_2$: C, 61.30; H, 4.41; N, 5.11. Found: C, 61.15; H, 4.51; N, 5.15.

9,10-Anthracenedibenzenesulfonamide.—To a solution of 9 g. of the crude adduct just described in 70 ml. of hot glacial acetic acid was added 6 drops of concentrated hydrochloric acid. Pale yellow crystals separated gradually and the mixture boiled from the heat of reaction. After warming on the steam-bath for one-half hour and then cooling, the product was collected and washed first with glacial acetic acid, then with cyclohexane; after drying it weighed 7.56 g. (95%), m.p. 243–244° (dec.). The product was dissolved in 5% aqueous sodium hydroxide (Darco), and precipitated by pouring into dilute hydrochloric acid. Recrystallization from pyridine–ethanol gave small yellow

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[6] L. F. Fieser, "Experiments in Organic Chemistry," D. C. Heath and Company, Boston, Mass., 1941, p. 345.

[7] O. Wichterle and M. Hudlický, *Collection Czechoslov. Chem. Communs.*, **12**, 564 (1947); H. Hagemeyer and D. Hull, *Ind. Eng. Chem.*, **41**, 2920 (1949).

needles, m.p. 243–244° (dec.). The infrared spectrum showed the presence of the -NH- linkage (3297 cm.$^{-1}$) and no carbon–nitrogen double bond.

Anal. Calcd. for $C_{26}H_{20}N_2O_4S_2$: C, 63.91; H, 4.13; N, 5.73. Found: C, 63.98; H, 4.18; N, 5.92.

Oxidation and Hydrolysis of 9,10-Anthracenedibenzenesulfonamide.—A mixture of 1 g. of 9,10-anthracenedibenzenesulfonamide suspended in 25 ml. of acetic acid and 1.65 g. of hydrated ferric chloride was heated on the steam-bath for one hour during which time no noticeable reaction took place. Upon the addition of 1.65 g. more of ferric chloride and refluxing the solid went into solution (30 minutes). The long light yellow needles that separated on cooling weighed 0.36 g. The acetic acid filtrate was poured into 3 *N* hydrochloric acid and the light yellow precipitate collected and dried. It weighed 0.05 g. The total yield was 0.41 g. (95%). Recrystallization from 100 ml. of absolute ethanol gave long light yellow needles, m.p. 285–286°. A mixture of this compound and anthraquinone melted at 285–286°.

9,10-Anthraquinonedibenzenesulfonimide.—To a suspension of 1.0 g. of 9,10-anthracenedibenzenesulfonamide in 20 ml. of glacial acetic acid was added 2 g. of lead tetraacetate. The mixture was heated to the boiling point then on the steam-bath for 14 hours during which time the diamide gradually went into solution. While still hot, 1 ml. of ethylene glycol was added and the solution filtered to remove a small amount of unreacted starting material. The filtrate on cooling deposited small lemon yellow prisms. The yield was 0.66 g. (66%), m.p. 225–231° (dec.). An additional 0.25 g. was obtained by pouring the acetic acid filtrate into water. Recrystallization from acetonitrile gave long pale yellow needles which melted at 240–241° (dec.), after turning yellow at 140–150°. This compound contained one molecule of acetonitrile of crystallization.

Anal. Calcd. for $C_{26}H_{16}N_2O_4S_2 \cdot CH_3CN$: C, 63.74; H, 4.01; N, 7.97. Found: C, 63.89; H, 4.25; N, 7.97.

Recrystallization from acetic acid gave small lemon yellow prisms, m.p. 240–241° (dec.). The infrared spectrum indicated the presence of a carbon–nitrogen double bond (1608, 1567 cm $^{-1}$).

Anal. Calcd. for $C_{26}H_{18}N_2O_4S_2$: C, 64.18; H, 3.73; N, 5.76. Found: C, 64.18; H, 3.97; N, 5.89.

To a suspension of 1.0 g. of 9,10-anthracenedibenzenesulfonamide in 20 ml. of glacial acetic acid was added 1.42 g. of sodium bismuthate. The mixture was refluxed for 30 minutes and filtered while hot. The residue weighed 0.33 g. and proved to be unreacted diamide. The filtrate was cooled and the crystals that had formed were collected, washed with glacial acetic acid and dried. The crystals weighed 0.28 g. (42%). Recrystallization from acetonitrile gave pale yellow needles, m.p. 240–241° (dec.).

To a suspension of 3 g. of 1,4-dihydro-9,10-anthraquinonedibenzenesulfonamide in 60 ml. of glacial acetic acid was added 12 g. of lead tetraacetate. The mixture was refluxed for 15 minutes during which time the compound went into solution. The clear pale orange solution was cooled, 10 ml. of ethylene glycol added, and the solution allowed to stand overnight at room temperature. The yellow prisms that had separated were collected on a filter, washed with 2 ml. of glacial acetic acid and dried. They weighed 1.33 g., m.p. 228–230° (dec.). Diluting the acetic acid filtrate with water gave an additional 1.48 g. of the same compound. The total yield was 2.81 g. (94%). Recrystallization from acetonitrile gave pale yellow needles, m.p. 240–241° (dec.). This product was identical with that prepared by the oxidation of 9,10-anthraquinonedibenzenesulfonamide.

Reduction of 9,10-Anthraquinonedibenzenesulfonimide.—A solution of 0.50 g. of 9,10-anthraquinonedibenzenesulfonimide in 25 ml. of tetrahydrofuran was treated with 1 g. of sodium hydrosulfite in 5 ml. of water. The two phase solution was boiled for one minute during which time a pale lemon yellow precipitate formed. The product was collected on a filter. It weighed 0.33 g. (66%) and proved to be 9,10-anthracenedibenzenesulfonamide, m.p. 242–243° (dec.).

Hydrolysis of 9,10-Anthraquinonedibenzenesulfonimide.—To a suspension of 0.55 g. of 9,10-anthraquinonedibenzenesulfonimide in 10 ml. of glacial acetic acid was added 5 drops of concentrated hydrochloric acid. The mixture was boiled for 5 minutes during which time the compound gradually went into solution. After cooling, the pale yellow needles that had separated were collected by filtration. They weighed 0.17 g., m.p. 285–286°. The filtrate was poured into water when 0.05 g. more of the same compound was obtained. The total yield was 93%. This proved to be anthraquinone.

Oxidation and Hydrolysis of 1,4-Dihydro-9,10-anthracenedibenzenesulfonamide.—To a suspension of 2.0 g. of 1,4-dihydro-9,10-anthracenedibenzenesulfonamide in 30 ml. of boiling glacial acetic acid was added 10 g. of sodium bismuthate in small portions. After the addition was completed (30 minutes), the solution was filtered while hot to remove a small amount of unreacted starting material (0.10 g.). After standing overnight the long orange needles that had separated from the filtrate were collected and washed first with water, then with ethanol. The yield was 0.37 g. (46%). Recrystallization from glacial acetic acid gave light yellow needles of anthraquinone, m.p. 285–286°.

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