

UNITED STATES PATENT OFFICE

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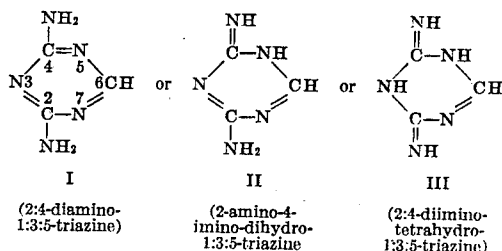
MANUFACTURE OF 2:4-DIAMINO-1:3:5-
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Geigy A. G., Basel, SwitzerlandNo Drawing. Application March 13, 1942, Serial
No. 434,624. In Switzerland April 25, 1939

10 Claims. (Cl. 260—249.5)

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This application is a continuation-in-part of my U. S. patent application Ser. No. 327,521, filed on April 2, 1940, now abandoned.

It is known that by heating fatty acid guanidine salts 2:4-diamino-1:3:5-triazines, such as for example formoguanamine or the diamino-cyanurohydrogen of the formula



can be obtained from guanidine formate or a mixture of guanidine sulfate and alkali formate. According to this reaction the homologous guanamines are also obtainable. As the last one the oenanthylic acid still gives a guanamine, but at the higher fatty acids however the reaction occurs in another direction. The yield, as indicated in the literature, cannot be said to be good. From 50 parts of guanidine salt only 5 parts of guanamine are obtained on the average.

Subsequent work has shown that the above mentioned reaction proceeds by way of the diguanide and that accordingly the guanamines can also be made by heating from fatty acid diguanide salts. In this case also the reaction does not take place smoothly, since secondary reactions (formation of ammeline and so on) may become the main reaction, as is known for the formation of acetoguanamine. The reaction of diguanide with fatty acid esters, e. g. formic acid ethyl ester—is it true—applicable for the manufacture of formoguanamine, but for the formation of the higher homologues it fails as well as the above mentioned processes.

Finally it is still known to interact diguanide with benzoylchloride in the presence of caustic soda. It has been found also in this case that the yields of phenylguanamine are extremely bad.

The other reactions for the formation of the guanamines may be disregarded, since they all start from expensive, complicated compounds, such as amidines, nitriles and so forth, and are therefore not interesting.

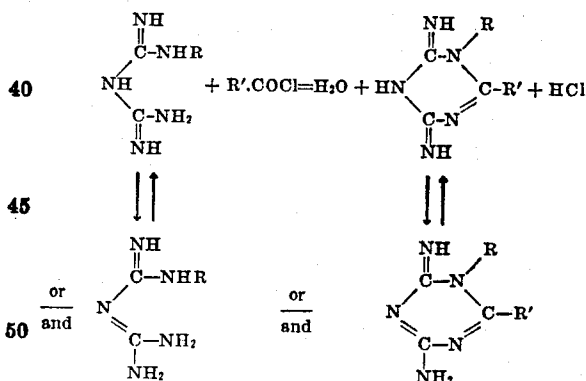
Now it has been found that surprisingly the 2:4-diamino-1:3:5-triazines are obtainable very readily and in a smooth manner by the reaction of diguanides with carboxylic acid-halides in

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presence of non-caustic alkalis. Thus, the higher homologues, which until now have not been accessible by interaction of fatty acids with guanidines, as well as a series of new substitution products are easily obtainable by a special choice of the diguanide derivatives and of the carboxylic acid chlorides.

The reaction of the cyanoguanidines, which, if desired, may be substituted, with the carboxylic acid halides generally proceeds relatively easily; it also begins when using relatively high molecular initial products already at temperatures of about 100° C. and proceeds at somewhat higher temperatures in the most cases practically completely, contrarily to the known processes, without formation of disturbing by-products. The products themselves are constitutionally determined by the kind of their synthesis; they are uniform.

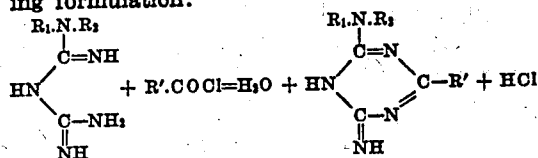
It has been found that from the different substitution products of cyanoguanidine, such as the alkyl- or aryl- or aralkyldiguanides and so on, and from carboxylic acid halides with more than one C-atom compounds are obtained, which are doubtless 5:6-di-alkyl-2:4-diimino-tetrahydro-1:3:5-triazines, 5:6-di-aryl-2:4-diimino-tetrahydro-1:3:5-triazines and 5:6-di-aralkyl-2:4-diimino-tetrahydro-1:3:5-triazines, as well as compounds with two different substituents in the positions 5 and 6, for example alkyl and aryl or aryl and aralkyl groups and so on. They are obtained from the said initial products by the action of carboxylic acid halides, especially the chlorides, under sufficiently energetic conditions, in presence of a hydrohalogen-binding, non-caustic agent. The reaction proceeds probably according to the following scheme:



wherein R and R' mean the same or different alkyls, aryls, aralkyls, other homocyclic or heterocyclic radicals, homocyclic- or heterocyclic-substituted alkyls or aryls with amino-, alkylamino-

or dialkylamino groups, alkyls and aryls with alkyloxy-, carboxylic, ether groups, also hydrocarbon radicals substituted or linked by other hetero-atoms than O and N.

Especially valuable compounds with improved properties are obtained by using as diguanide derivative an aryl- or aralkyl-alkyldiguanide containing aryl or aralkyl and alkyl groups at the same nitrogen atom. Preferably the starting compounds are prepared by causing alkyl arylamines or alkyl aralkylamines respectively to react with dicyano diamide. In this case the ring-closing takes place according to the following formulation:



wherein R_1 and R_2 mean aryl, aralkyl and alkyl radicals.

The new compounds may serve as intermediate products for the manufacture of medicinal and therapeutic substances and dyestuffs, but also as textile assistants, such as for example in the vat-dyeing, as softening agents and so on.

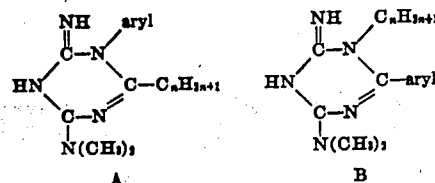
From the technical point of view compounds of this group, which contain at least one higher non-aromatic hydrocarbon radical, e. g. the alkyl group of a saturated or an unsaturated fatty acid or their mixtures, the hydrocarbon radical of a hydroaromatic acid, of resin acid, naphthenic acid, whereby this substituent may occupy the 5- as well as the 6-position or in some cases both positions may be occupied in such manner, are particularly interesting.

It is also important in respect of the technical use of the products, that they can be transformed into the most various derivatives for the purpose of graduation or variation of the properties without splitting up the hetero-cycle. The aryl- and aralkyl derivatives can be nitrated and the nitro derivatives reduced; they can also be sulfonated, whereby the sulfonating degree can be adjusted to the desired solubility. The sulfuric acid radical or the SO_3Na -group can also be introduced without the intermediate of an aryl or aralkyl by treating a hydroxyalkyl derivative with sulfuric acid or chlorosulfonic acid or by interacting a chloralkyl derivative with sulfite.

Furthermore, one or more hydrogen atoms bound to nitrogen can be replaced by alkyl, hydroxyalkyl, halogenalkyl, aminoalkyl, chlorhydroxyalkyl and so on; then the tertiary amino groups can be converted by addition of halogenalkyl, dialkylsulfate, ethylene oxide and other alkylene oxides in presence of water into quaternary ammonium groups. When the 6-position is occupied by a methyl- or a methylene group it can be condensed with aldehydes, especially with aromatic aldehydes or their substitution products, thus enabling further reactions.

As a further possibility of variation it may be mentioned that, generally in all cases and practically in most cases, by a simple interchange of the substituents in 5- and 6-position every combination is obtainable in form of two compounds isomeric as to position. Thus it is, for example, possible to obtain by condensation of aryldiguanide with fatty acid chloride and after-treating with dimethylsulfate and soda the compound according to Formula A, and from alkyldiguanide with arylcarboxyl acid chloride and after-treat-

ing as above the isomer compound according to Formula B:



The invention is illustrated, but not limited, by the following examples, the parts being by weight, except as otherwise indicated.

Example 1

36 parts of phenyldiguanide are dissolved in 400 parts of toluene, 24 parts of anhydrous soda (sodium carbonate) are added and gradually combined under stirring at 50°-60° C. with 42 parts of lauric acid chloride dissolved in the same quantity of toluene. The temperature is raised gradually to 110° C., and is then kept thereat for 16 hours, then the whole is allowed to cool down to 80° C., diluted with alcohol, the solution is filtered off from the salt residue and concentrated in vacuo. There is obtained without further purification the 2:4-diimino-5-phenyl-6-undecyl-tetrahydro-1:3:5-triazine in form of white, yellowish crystals, which are soluble in mineral and organic acids and which again precipitate on diluting. The yield amounts to 67 parts instead of 72 parts calculated, i. e. to 93% of the theory. The use of tertiary amines instead of soda (sodium carbonate) affords no advantage.

If instead of a single acid chloride the mixture of the chlorides obtained from palm nut fatty acid is used, a product of a yield of about 96% is obtained, which hardly can be differentiated as to constitution and properties from that obtained from lauric acid. Likewise the production with the acid chlorides of the fish-oil fatty acids or other fatty acids from natural fats, oils, waxes and so on takes place very easily.

In the same manner as with phenyldiguanide the acid chloride reacts with diguanide, methyl- and butyldiguanide, chlorophenyl-, o- and p-toluy-, anisidyl-, cyclohexyl- and pyridyldiguanide. Also nitroaryl- and aminoaryldiguanides as well as amino- and alkylamino-alkyldiguanides such as for example diethylaminoethylidiguanide react in sufficient degree.

Example 2

120 parts of stearic acid chloride diluted with the same quantity of chlorobenzene are introduced, while well stirring at 50° C., gradually into a solution of 71 parts of phenyldiguanide in 800 parts of chlorobenzene after having added 45 parts of soda (sodium carbonate). The temperature suddenly rises to 100° C. and is then raised to 110-115° C., and the whole mixture is kept at this temperature for 14 hours. Then it is diluted at about 80° C. with the half volume of methanol, filtered and concentrated in vacuo. There is obtained the 2:4-diimino-5-phenyl-6-heptadecyl-tetrahydro-1:3:5-triazine in calculated yield in form of nearly pure white crystals which after recrystallisation melt at 113-114° C. This compound dissolves in organic solvents, but not in water; acids do not dissolve it, but disperse it in fine flakes.

The heptadecenyl- or naphthenyl derivatives made in the same manner from phenyldiguanide and oleic acid or naphthenic acid (neutr. number 215) are liquid, but in their behaviour they hardly

differ from the derivatives of the other acids. The other diguanides, enumerated at the end of Example 1), give similar compounds.

Example 3

100 parts of the product obtained according to Example 2 are introduced at about 30° C. within ½ hour into 200 parts of sulfuric acid monohydrate, then after dissolution being finished 100 parts of oleum of 26 per cent strength are added and the whole is still stirred for 1½ hours. Then the mass is stirred with a water-ice-mixture, the aqueous layer is separated off, the residue is pasted with water, then neutralized with caustic soda lye and the solution concentrated by evaporation. There remain 120 parts (calculated 124 parts) of sulfonate as a greyish-white crystal meal. The sulfonate dissolves clearly in water, the solutions are precipitated by sulfuric acid.

For the manufacture of a sulfonic acid soluble also as free acid in water or in an excess of mineral acid stronger conditions are needed. In this case for example 100 parts of the substance are dissolved in a mixture of 200 parts of sulfuric acid and 100 parts of oleum of 26 per cent strength, the whole is stirred for 10-12 hours at about 35° C., then 100 parts of oleum are further added and the whole is stirred for six further hours, whereupon the sulfuric acid in excess is separated and the neutralized reaction product isolated as above stated. The same is a grey crystal-meal which becomes nearly white by decoloring with decoloring carbon; it readily dissolves in water and does not precipitate on addition of sulfuric acid and so on.

Example 4

10 parts of the product obtained according to Example 2 are heated under reflux in 100 parts of toluene with 3 parts of dimethyl sulfate and 5 parts of anhydrous soda (sodium carbonate) for 5 hours, and it is then filtered and the filtrate concentrated in vacuo. The residue is heated with the equimolecular quantity of dimethyl sulfate for ½ hour on the water-bath. The product forms a thick, white paste, which dissolves in water to an opalescing liquid which becomes clear, on addition of acetic acid, becomes turbid by acids and is again precipitated by bases.

If the intermediate product is treated with the double quantity of dimethyl sulfate (i. e. molecular proportion 1:2) and soda under the same conditions, if the methyl sulfate of sodium and the soda in excess is separated off and if dimethyl sulfate is again caused to react without addition of soda, the thus obtained product is nearly completely soluble in water to an only weakly opalescing solution even in the presence of mineral acid. Instead of using dimethyl sulfate the methylated compound can also be made quaternary by means of ethylene oxide or epichlorhydrine by digesting, then heating the aqueous or aqueous-alcoholic solution, which may be acidified. The compound with the equimolecular quantity of stearic acid is turbidly soluble in water.

Instead of the compound used in the above example any one of the many other compounds cited in the Examples 1 and 2 may also be employed.

Example 5

The product made according to Example 1 is dimethylated with double the equimolecular quantity of dimethyl sulfate and a small excess of soda, as described in Example 4 for the heptadecyl derivative. Then the double quantity by weight of sulfuric acid and the same weight of

oleum of 26 per cent strength is caused to react therewith and the reaction mixture is worked up as described in Example 3. Thus there is obtained the sodium salt of the dimethyl-2:4-dilmino-6-undecyl-5-phenylsulfonic acid. It forms a greyish-white crystal-powder, which is clearly soluble in water. Sulfuric acid precipitates the free acid.

If the alkylation is carried out by means of diethylsulfate or another of the above mentioned compounds as initial material, similar products in their appearance and behaviour are obtained.

Example 6

A solution of 3 parts of fencholic acid chloride in 4 parts of toluene is caused to drop in the well stirred mixture, heated to 60° C., of a solution of 3 parts of phenyldiguanide (or equivalent parts of another of the above said diguanides) in 20 parts of toluene and of 2 parts of soda. The temperature increases to about 100° C., and is then raised to 120° C. and kept at this level for several hours. Then further 3½ parts of soda are added and 4 parts of dimethylsulfate are caused to drop in. After heating for 5 hours under reflux the solution is filtered from the salt paste and the filtrate is freed in vacuo from the solvent. The residue, a yellow, soft-resin-like mass, is then dissolved at 15°-20° C. in the double quantity by weight of sulfuric acid, thereupon the same quantity of oleum of 25 per cent strength is gradually added, the whole is stirred for 3 hours and the resulting sodium salt of dimethyl-2:4-dilmino-6-(1'-methyl-3'-isopropyl)-cyclopentyl-1:3:5-triazine-6-phenylsulfonic acid is isolated as described in Example 3 for the analogous heptadecyl derivative.

Instead of the fencholic acid chloride the bromide or another acid halide, such as for example the halide of acetic-, butyric-, valeric-, caproic-, caprylic-, palmitic acid and so forth may be used. Instead of soda the carbonates or bicarbonates of the alkali- or earth-alkali metals or other non-caustic alkalies can be used in the above examples.

Example 7

117-118 parts of stearic acid chloride diluted with 100 parts of chlorobenzene are allowed to drop into a solution of 79 parts of dry methyl phenyldiguanide in 500 parts of chlorobenzene, which is stirred at 70° C. with 50 parts of anhydrous sodium carbonate, whereby the temperature rises up further to 10°-20° C. The whole is heated up to 110°-120° C., stirred for about 20 hours, whereupon the still hot solution is separated from the salt paste by means of a centrifuge or by suction. Now the solution is stirred with 100 parts of anhydrous sodium carbonate, then 104-105 parts of dimethyl sulfate are allowed to gradually run thereinto at 50° C., the whole is heated up to 110°-120° C. for further 5 hours, while continuously stirring, centrifuged or sucked off and the chlorobenzene distilled off in vacuo. Thus, there remain 187 parts, i. e. 97% of the calculated quantity of the methylated compound.

The total quantity is stirred or kneaded at 0°-5° C. with 400 parts of sulfuric acid monohydrate, care being taken that the temperature does not rise above 10°-20° C. Then at a maximum of 20°-25° C. 200 parts of fuming sulfuric acid (containing 26% of trioxide) are gradually added thereto. The whole is stirred for several hours until a test, after neutralisation, is clearly soluble in water, and the mass is introduced into 1500 parts of ice, whereupon, if neces-

sary, water is added, until a complete separation of the layers has been reached, and finally the diluted sulfuric acid is separated from the sulfonic acid remaining in an undissolved condition.

The sulfonic acid layer is stirred with water (according to the desired percentage of the final product with the equal or manifold quantity by weight), then 50 parts of anhydrous sodium carbonate are added thereto thus separating for the greatest part the sulfuric acid affixed to the amino groups and the whole is completely neutralised by means of concentrated caustic soda lye.

The compound constitutes a yellowish crystal meal which with a little water swells jelly-like, but is clearly soluble in some more water. The solution is stable towards bases and salts (the water-hardening agents). It shows excellent capillary-active properties and is suitable for various technical purposes, especially useful as textile assistants such as for example as stripping and levelling agent in vat-dyeing.

The methylphenyldiguanide serving as starting material may be prepared as follows:

107 parts of monomethylaniline are dissolved in a mixture of 100 parts of concentrated hydrochloric acid and 400 parts of water. Then 92 parts of dicyanodiamide are added thereto, the whole is heated for 2 hours, while stirring, up to 105° C. and stirring continued at this temperature for further 18 hours. After cooling down, 140 parts of caustic soda lye of 30% strength, previously diluted with 250 parts of water, are allowed to gradually flow therein and the solution, which has been warmed up to about 35° C. is stirred for half an hour, whereupon the reaction product is precipitated out by the addition of 150 parts of sodium chloride. After exhausting by suction and drying of the precipitate there are obtained about 170 parts of N-methyl-N-phenyldiguanide, that is to say a yield of about 90% of the theory.

Instead of pure methyl aniline there can also be used a raw mixture of mono- and dimethylaniline resulting from the methylation of aniline. Thus for example instead of 107 parts of methylaniline 267 parts of a mixture of 60% of dimethyl- and 40% of monomethylaniline are worked up, as above indicated, in a hydrochloric solution with dicyanodiamide and the reaction mixture is then maintained at a temperature of 70°-80° C. until the separation in two layers has taken place. The upper layer which is now separated consists of 155-160 parts of dimethylaniline which thus is isolated in a yield of 97-98% of the theory. The lower aqueous layer contains the methylphenyldiguanide hydrochloride which is separated out in the same manner and with the same yield as above indicated, by means of caustic soda lye and sodium chloride.

Example 8

41 parts of N-ethyl-N-phenyldiguanide or the corresponding quantity of the dry precipitate containing sodium chloride, as obtained according to the indications given below, diluted with manifold the weight of chlorobenzene are caused to react in the presence of 24 parts of anhydrous sodium carbonate first at 70°-80° C., finally at 110°-120° C. with 56 parts of stearic acid chloride; then it is filtered from the salt paste and, after the addition of 45 parts of anhydrous sodium carbonate, the filtrate is treated with 50 parts of dimethyl sulfate whereby the temperature of initially 50° C. is finally increased to 110° C. After distillation of the chlorobenzene in

vacuo, there remain 88 parts (calculated 92 parts) of dimethyl derivative.

For the sulfonation this derivative is dissolved in the double quantity by weight of sulfuric acid of 100% strength and treated with oleum containing 26% of SO₃, until a sample has become soluble in caustic alkali. The usual working up yields more than 100 parts of sulfonate which, with regard to appearance and solubility, hardly differs from the product obtained according to Example 7.

The starting material can be prepared as follows:

240 parts of monoethylaniline dissolved in a mixture of 200 parts of concentrated hydrochloric acid and 700 parts of water are stirred for 20 hours at 105° C. with 184 parts of dicyanodiamide. After cooling, 320 parts of caustic soda lye of 30% strength diluted with 500 parts of water are added thereto. By precipitating by means of 500 parts of sodium chloride, centrifuging and drying there are obtained about 350 parts of N-ethyl-N-phenyldiguanide.

In the same manner as monoethyl- and monomethylaniline the N-ethyl- and N-methyltoluidines, the N-alkylxylyldines, -anisidines, -phenetidines and other alkylarylamines substituted in the aromatic nucleus can also be condensed with dicyanodiamide and the diguanide derivatives thus obtained be caused to react first with acid chloride, then with dialkylsulfate and finally be sulfonated.

The conversion of the alkyl-aralkyl-diguanides with acid chlorides and the further production of derivatives are carried out in the same manner. However, in these cases the preparation of the starting products by the reaction of the amine hydrochloride with dicyano diamide is often not possible or not productive. The starting products may then be produced analogously to the preparation of dialkyl-diguanides by way of the copper complex salts. For example, dicyanodiamide gives, in an aqueous alcoholic medium in the presence of the equimolecular quantity of copper sulfate, with an excess of ethyl- (or methyl-) benzylamine (obtained by a condensing hydrogenation of benzaldehyde with ethylamine, B. P.₁₂ mm. 110°-112° C.) the complex salt from which the copper is eliminated in a known manner by means of hydrogen sulfide, while the N-ethyl- (or N-methyl-)N-benzylidiguanide is isolated.

Example 9

57 parts of the methylphenyldiguanide obtained according to Example 7 are condensed in the presence of 45 parts of anhydrous sodium carbonate with 60 parts of the chlorides of the palm nut fatty acid. The intermediate product is treated first with 76 parts of dimethyl sulfate (i. e. two molecules per one molecule of the substrate) in the presence of 90 parts of anhydrous sodium carbonate, then, after the separation of the resulting sodium methylsulfate and of excess sodium carbonate, with a further quantity of 76 parts of dimethylsulfate without addition of sodium carbonate. After having expelled the diluent such as e. g. chlorobenzene in vacuo the final product is obtained in form of a semi-solid gel. It is completely clearly soluble in water and the highly capillary-active solution is stable towards acids, while by the addition of bases there is caused a turbidity or precipitation respectively.

When in the second (last but one) reaction phase, the alkylation in the presence of sodium

carbonate, the dimethyl sulfate is replaced by the equivalent quantity, that is to say by 92 parts of diethyl sulfate, one obtains by the subsequent treatment with dimethyl sulfate alone an equivalent final product which is clearly soluble in water. If, however, in the last reaction phase the dimethyl sulfate is replaced by diethyl sulfate, the final product gives with water an opalescing solution and this independently of whether in the last but one phase there has been used dimethyl sulfate or diethyl sulfate.

If in the first reaction phase there is used technical stearic acid chloride and if the resulting intermediate product is worked up, as above indicated, with dimethyl or diethyl sulfate first in the presence and then in the absence of sodium carbonate, the final product is, except for the somewhat greater consistency, equivalent to the product prepared by means of palm nut fatty acid chloride. When using the chlorides of the cocos acids, it is, however, more difficult to obtain products which are clearly soluble in water.

Example 10

70 parts of the ethylphenyldiguanide prepared according to Example 8 are dissolved in chlorobenzene, then 45 parts of anhydrous sodium carbonate are added thereto and about 65 parts of palm nut fatty acid chloride are allowed to flow thereinto at 70° C., whereupon the reaction is completed by gradually increasing the temperature up to 110°-120° C. and the intermediate product is isolated.

By the reaction of first 76 parts of dimethyl sulfate in the presence of 90 parts of anhydrous sodium carbonate and of a further equal quantity of dimethyl sulfate without addition of sodium carbonate a product is obtained which with regard to its nature and behaviour, especially with respect to complete water-solubility, resembles the product obtained according to Example 9.

Also in this case the dimethyl sulfate may be replaced in the last but one reaction phase by the corresponding quantity of diethyl sulfate without impairing the solubility of the final product, while the use of diethyl sulfate in the last phase gives products whose aqueous solutions are turbid.

Example 11

152 parts of methylphenyldiguanide are caused to react with 180 parts of naphthenic acid chloride in the presence of 120 parts of anhydrous sodium carbonate. The intermediate product is treated with 190 parts of dimethyl sulfate or with the corresponding quantity of diethyl sulfate in the presence of 200 parts of anhydrous sodium carbonate, then 190 parts of dimethyl sulfate are caused to react alone with this mixture, whereby throughout all of the phases the conditions mentioned in the Examples 7 and 8 are observed. The final product constitutes a clear gel which with water gives weakly opalescing solutions.

Example 12

Methylphenyldiguanide is condensed with the equimolecular quantity of fencholic acid chloride and twice two-fold the equimolecular quantity of dimethyl sulfate, first in the presence and then in the absence of sodium carbonate, is caused to react with the intermediate product. Thus there is obtained a product giving with water strongly opalescing solutions.

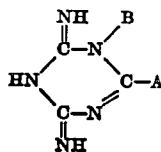
Example 13

95 parts of methylphenyldiguanide are con-

densed according to Example 7 with 135 parts of technical stearic acid chloride and sodium carbonate. The intermediate product is dissolved in alcohol of 90% strength, treated with 92 parts of epichlorhydrine and heated in the autoclave for several hours up to 120°-130° C., whereupon the alcohol is expelled. The residue (about 220 parts) solidifies after some time in a crystalline form. It shows no more reaction for chlorine ions. By the interaction of 63 parts of dimethyl sulfate the said residue is converted into a moderately viscous liquid which gives with water weakly opalescing solutions.

What I claim is:

1. A process for the manufacture of derivatives of 2:4-diamino-1:3:5-triazines, comprising causing a diguanide selected from the group consisting of diguanide, a monoalkyl substituted diguanide, a monoaryl substituted diguanide of the benzene series, an alkylaryldiguanide of the benzene series and an alkylbenzyldiguanide to react with a carboxylic acid halide in presence of a hydrohalogen-binding agent selected from the group consisting of the alkali metal carbonates, alkaline earth metal carbonates, alkali metal bicarbonates and alkaline earth metal bicarbonates.
2. A process as claimed in claim 1, wherein the reaction components are so chosen that the final product contains at least one organic substituent with more than 6 C-atoms in the 5-position.
3. A process as claimed in claim 1, wherein the reaction components are so chosen that the final product contains at least one organic substituent with more than 6 C-atoms in the 6-position.
4. A process for the manufacture of a 2:4-diamino-1:3:5-triazine derivative, comprising reacting a diguanide selected from the group consisting of diguanide, a monoalkyl substituted diguanide, a monoaryl substituted diguanide of the benzene series, an alkylaryldiguanide of the benzene series and an alkylbenzyldiguanide to react with stearic acid chloride in presence of an alkali metal carbonate.
5. A process for the manufacture of a 2:4-diamino-1:3:5-triazine derivative, comprising reacting a monoaryldiguanide of the benzene series with a carboxylic acid halide containing at least 6 carbon atoms in presence of an alkali metal carbonate.
6. A process for the manufacture of a 2:4-diamino-1:3:5-triazine derivative, comprising reacting phenyldiguanide with stearic acid chloride in presence of an alkali metal carbonate.
7. A process for the manufacture of a 2:4-diamino-1:3:5-triazine derivative, comprising reacting phenylmethyldiguanide with stearic acid chloride in the presence of an alkali metal carbonate.
8. As new compound 2:4-diimino-5-phenyl-6-heptadecyl-tetrahydro-1:3:5-triazine, being white crystals, melting at 113-114° C., soluble in organic solvents, insoluble in water.
9. The sodium salt of a sulphonic acid of 2:4-diimino-5-phenyl-6-heptadecyl-tetrahydro-1:3:5-triazine, being a substantially white crystal meal, soluble in water.
10. The 2:4-diamino-1:3:5-triazine derivatives of the following general formula



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wherein A means an aliphatic radical with more than 6 carbon atoms and B means an aromatic radical of the benzene series.

ADOLF GRÜN.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,217,030	Simons	Oct. 8, 1940
2,258,130	Brunson	Oct. 7, 1941

Number
2,202,828
2,270,478
1,549,901

12

Name	Date
Brunson	June 4, 1940
Schmidt	Jan. 20, 1942
Bonhote	Aug. 18, 1925

FOREIGN PATENTS

Number	Country	Date
466,096	Great Britain	1937

OTHER REFERENCES

Annalen 376, pp. 167, 180, 181.
 Berichte de Deut. Gess. 24, pp. 2594-2601.
 J. Prakt. Chem. 2 (84), p. 403.