

The decomposition of organic ...

S/056/02/000/000/010/011
E194/E135

relationships between the yields of individual gases, for example acetylene, from the various organic liquids were very similar with the two kinds of discharge. Calculations similar to those made in the previous article were repeated, and again agreement with experiment was good. It was found that increasing the impulse duration from 10^{-6} to 10^{-3} seconds increases the yield of acetylene from unsaturated hydrocarbons by 5-6%, whilst the use of an a.c. arc described in the previous work raises it a further 3-5%. Decomposition of liquid hydrocarbons in an arc theoretically requires less energy to produce a given volume of acetylene than when pulses are used, and the yield is higher with an arc. The data obtained are valid only for the particular test conditions. There are 1 figure and 4 tables.

Card 2/2

PERSIN, S.M.

Methods for increasing the precision of coding devices by automatic elimination of coding errors. Trudy GGO no. 112: 14-32 '63.

Principles of functional conversion of information using piecewise linear approximation of functions. Ibid.:33-57

Functional coding converters using the principle of control of the pulse frequency. Ibid.:58-73 (MIRA 17:5)

PESIN, V. G.

Synthesis of plazthiole (2, 1, 3-benzothiadiazole) and its derivatives. A. M. Khaletskii and V. G. Pesin (Leningrad Chem.-Pharm. Inst.). Zhur, Obshchei Khim. (J. Gen. Chem.) 20, 1914-20(1950); of, Hinsberg, Ber, 22, 2895 (1889)—Heating 1 g. o-C₆H₄(NH₂)₂ and 7 ml. aq. NaHSO₃ (d. 1, 3; bisulfite content 32%, SO₂ content 20%) 6 hrs. to 190-200° in a sealed tube gave in extn. with C₆H₆ and evapn. of the ext. 88% plazthiole(I), m. 42°. Heating 50 g. o-C₆H₄-(NH₂)₂ and 150 g. SOCl₂ in 2200 ml. dry MePh 14-15 hrs. gave on distn. 84% I, b. 206°, m. 44°. I dissolves in concd. mineral acids, but the salts are hydrolyzed on diln. and the base is extractable with C₆H₆; I, HCl is insol. in concd. HCl; I and Ph NH₂ form a yellow soln. I is stable to HgCl₂ (eve) at 250° and is unchanged by K₂Cr₂O₄, H₂SO₄, although weakly acid KMnO₄ slowly oxidizes it, giving unknown----- Heating I with Zn-AcOH yields H₂S and C₆H₄(NH₂)₂;-----Ac₂O have no action on I. Nitration of I (25 g.)-----H₂SO₄ at 0° with 15 ml, HNO₃ (d, 1, 4) and----- followed by 0.5 hr. at 20° gave 90 3%-----of I, in. 103°. Reduction of this by re-----A-OH gave 80%-----forming an easily hydrolyzable HCl----- heating this with Zn-HCl gives H₂S and 1, 2, 5-C₆H₃-----heating the amino deriv. with - O₂NC₆H₄, COCl in gave the 4-nitro deriv., m. 226-7° (from EtOH); heating this 15 min. with 10% HI gave 4-nitro-1, 2-phenylenediamine, in 198° (from MePh), after neutralization by NH₄ OH-heating this (2 g.) with 4 g. SOCl₂ in dry MePh gave a product, m. 101-3° identical with I.

G. M. K.

PESIN, V. G.

USSR/Chemistry Synthesis

Card 1/1 Pub. 151 - 25/36

Authors : Khaletsky, A. M., and Pesin, V. G.

Title : Synthesis of piazothiole (3,4-benz-1,2,5-thiodiazole) and its derivatives. Part 2.-

Periodical : Zhur. ob. khim. 24/1, 133-136, Jan 1954

Abstract : The synthesis of 2-nitro, and 2-aminopiazothioles, 4-nitrophenyl-1-sulfamide-piazothiole, 4-aminophenyl-1-sulfamide-piazothiole, 4-nitrophenyl-2-sulfamide-piazothiole and 4-aminophenyl-2-sulfamide-piazothiole is described. The displacement of the hydrogen of the benzene ring in position 1 by the nitro group, was observed during the nitration of the piazothiole. The fact that nitro- and aminopiazothioles can exist in the form of two different isomers was confirmed experimentally. Three references: 1-USA and 2-USSR (1928-1953).

Institution : The Chemical-Pharmaceutical Institute, Leningrad

Submitted : September 26, 1953

V. Pesin, V.G.

Chemistry of ...
translation -- See C. I. 50, 1988e

PM

Pesin, V. G.

USSR/ Chemistry - Physical chemistry

Card 1/1 Pub. 22 - 23/43

Authors : Khaletskiy, A. M.; Pesin, V. G.; and Chzhao Chzhi-Chzhun

Title : Study of the chemistry of piaethiol (3,4-benz-1,2,5-thiodiazole)

Periodical : Dok. AN SSSR 106/1, 88-91, Jan 1, 1956

Abstract : A study of the chemical properties of piaethiol derivatives showed that the benzene ring in the heterocycle retains its aromatic nature. Chlorination, bromination and thiocyanation of 1- and 2-aminopiaethioles led to the formation of homologous mono- and di-chloro-bromo and thiocyanogen derivates. The properties of the thiourea derivatives obtained during the reaction of ammonium thiocyanate with hydrochloride-1 or 2-aminopiaetholes are described. The products of aminopiaethiol azotization in a sulfate medium are listed. Four references: 1 USSR, 1 Ital. and 2 Germ-USA (1889-1955). Table.

Institution : Leningrad Chemicopharmaceutical Institute

Presented by: Academician I. N. Nazarov, September 26, 1955

RESIN, V. G.

1
 V Chemistry of 2,1,3-thiadiazole. III. Reaction of aromatic diamines with acetyl chloride and thionyl chloride. V. D. Kuznetsov, A. M. Khachatryan and Chi-Chun Chao (Chem. Zhurnal, Leningrad Univ. Obshchei Khim. 27, 1570-61, 1967); cf. C.A. 49 4644. Heating 1.5 g 4-aminodiaminobenzene in C_2H_5Cl with 3 g $PhNCSO_2$ 2 hrs. on a steam bath gave 88.8% of 2,1,3-thiadiazole, m. 128° (lit. 128°), the yield was lower in $MeOH$ or $EtOH$ and slightly higher in $MePh$, but lower in $EtPh$. The 4-amino-1,2-diaminobenzene was prepd. in 88% yield (m. 103-5°) by addn. to the refluxing aq. soln. of 36 g 2,4-diaminodiamine in 150 ml. $MeOH$ with 30 g. NH_4Cl of 110 g. Na_2S over 1 hr., followed by heating 1 hr., sepn. of the red ppt. and heating with 200 ml. 4% HCl with subsequent sepn. of the unreacted starting material by filtration; addn. of NH_4OH to the filtrate gave the desired amine. Similarly, 23 g. *p*-toluylenediamine and 53 g. $PhNCSO_2$ in $MePh$ gave 89% 5-methylbenzo-2,1,3-thiadiazole, m. 84°. 4-Ethoxy-1,2-phenylenediamine gave 97% 5-ethoxybenzo-2,1,3-thiadiazole, m. 76-7° (aq. $EtOH$), while 4-chloro-1,2-phenylenediamine gave 84% 5-chlorobenz-2,1,3-thiadiazole, m. 52-5°. Similarly was prepd. 81% 5-bromobenz-2,1,3-thiadiazole, m. 61°. Et 3,4-diaminobenzoate gave 95% Et benzo-2,1,3-thiadiazole-5-carboxylate, m. 57-8.5°. 3,4-Diaminobenzoic acid gave 86.6% benzo-2,1,3-thiadiazole-5-carboxylic acid, m. 220°. 3,4-Diaminobenzenesulfonic acid in $PhNH_2$, treated with $PhNCSO_2$ as above gave after 3 hrs. at 120° a substance, m. 220-3°, apparently benzo-2,1,3-

Distr: AE43

PESTIN, V.G.; KHALITSKI, A.M.; CHAO, CHI-CHUN

thiadiazole-5-sulfonic acid, probably as the $PhNH_2$ salt; this with $BaCO_3$ gave the Ba salt which with H_2SO_4 gave 88% *benzo-2,1,3-thiadiazole-5-sulfonic acid*, m. 211-5° (lit.). *4,6-Diaminobenzoic acid* similarly gave 83.8% *5-hydroxybenzo-2,1,3-thiadiazole-6-carboxylic acid*, m. 211-5° (lit.). The reaction of *o*-diamines with $SOCl_2$ is slow but can be accelerated by addition of H_2N or pyridine, when yields of 85-95% of thiadiazoles are obtained. IV. Chlorination and bromination of *benzo-2,1,3-thiadiazole*. *Ibid.* 1875-81. It is shown that the statement by Efros and Levit (C.A. 50, 783) concerning the lack of reactivity of *benzo-2,1,3-thiadiazole* is incorrect. The substance is aromatic in character and does react under proper conditions. Thus, chlorination of *benzo-2,1,3-thiadiazole* in the presence of Fe shavings at 60° gave 4,7-dichloro deriv., m. 181-2.5°, after treatment of the crude product with 10% alc. KOH. Cooling the crude chlorination product in H_2O aq. gave up to 22% *tetrachloro-4,7-dichloro-2,1,3-thiadiazole*, m. 120-4°, and its isomer, m. 82°. If the Fe catalyst is omitted, chlorination at 60° gave the same products as above. Nitration of 2,3-dichloro-*benzo-2,1,3-thiadiazole* and reduction of the isomer mixt. gave homeric diamino-2,3-dichlorobenzenes; these (4.5 g.) in C_6H_6 treated with 4.5 g. $PhNSO$ and heated on a steam bath 0.5 hr gave after washing with 4% HCl 28.8% *4,5-dichloro-2,1,3-thiadiazole*, m. 112-13°. Similarly, *4,5-dichloro-1,2-diaminobenzene* gave 88.3% *5,6-dichloro-2,1,3-thiadiazole*, m. 112-13°.

1
2
1

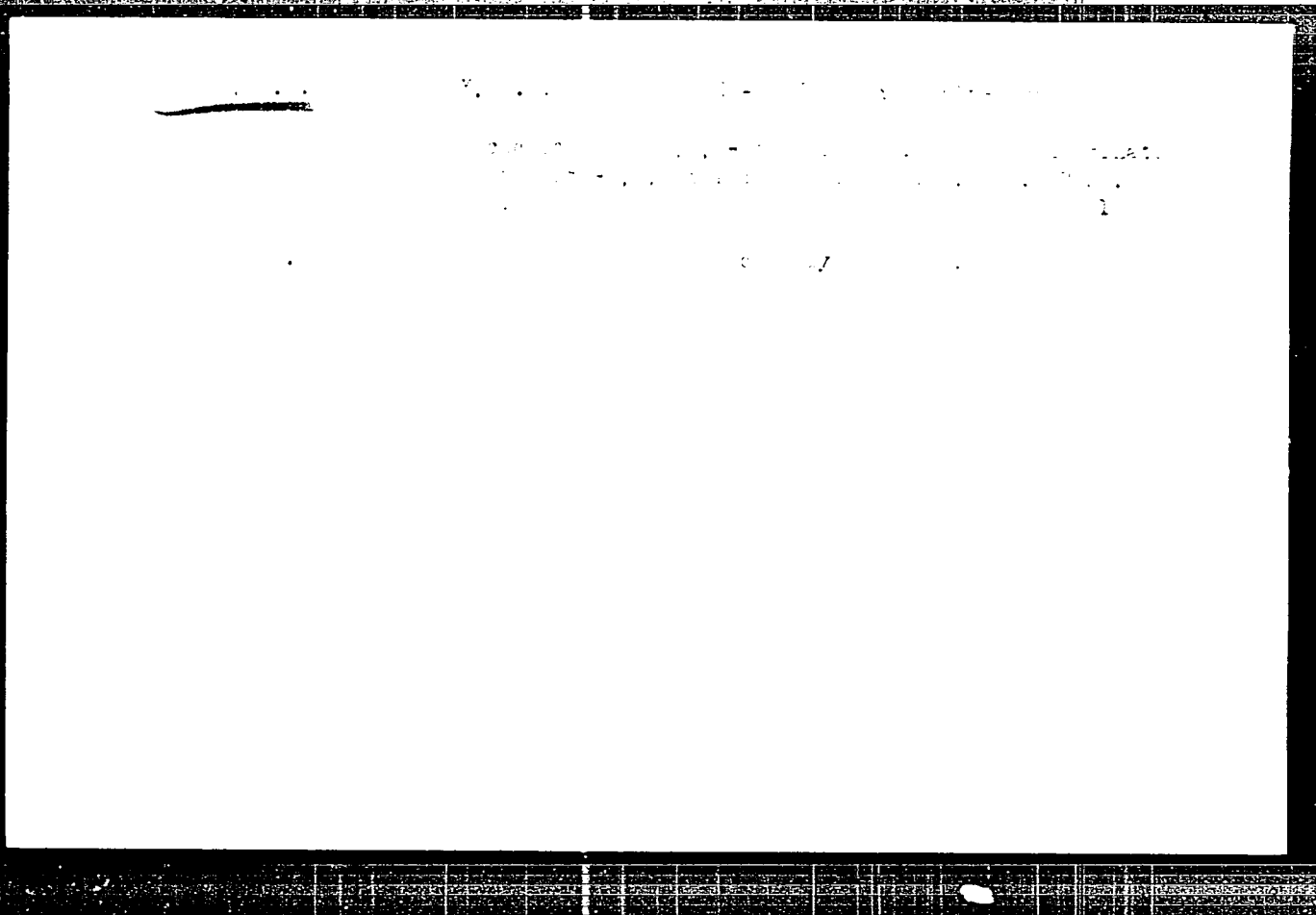
2/3

PESIN, V.G.; KHALETSKI, A.M.; CHAO, CHI-CHUN

Similarly, 4,6-dichloro-1,2-aminobenzene gave 96% 5,7-dichlorobenz-2,1,3-thiadiazole, m. 67-7.7°. Similarly was prep'd. 4,7-dichlorobenz-2,1,3-thiadiazole, m. 181-2.6°. Brominating 0.8 g benz-1,3-thiadiazole with 32 g Br at 65-77° and refluxing 1 m. gave 85.8% tetrabromodihydrobenzo-2,1,3-thiadiazole, m. 142°; this heated with aq. alc. KOH 1 hr. gave 80% 4,7-dibromobenz-2,1,3-thiadiazole, m. 104-5°. Heating 3.6 dibromo-1,2-aminobenzene with PhNSO in C₆H₆ gave the same substance, m. 181-5°. Thus, halogenation of the benzothiadiazole yields tetrahalotetrahydro deriv. which loses 2 mols HX on treatment with base and yields a dihalo-substituted thiadiazole. The substance, m. 67.6°, reported by H. and L. (*loc cit*) may be a dichlorodihydrobenzothiadiazole.

G. M. K.

3/3



PESIN, V.G.; KHALETSKIY, A.M.

Chemistry of 2,1,3,-thiodiazole. Part 5: Nitration of some mono-
and dihalogen derivatives of benz-2,1,3,-thiodiazole. Zhur. ob.
khim. 27 no.9:2599-2604 S '57. (MIRA 11:3)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Pyrrole) (Nitration)

15324, 111

PA - 3163

AUTHOR: KHALETSKIY, A.M., PESIN, V.B. and CHEHAZ CHZHI-CHZHUN
TITLE: Inquiries into the Chemistry of Plazothione (3,4-benz-1,2,5-thiodiazole).
(Issledovaniya v oblasti khimii plazotona (3,4-benz-1,2,5-tio diazola) Russian).
PERIODICAL: Doklady Akademii Nauk SSSR 1957, Vol. 113, Nr 3, pp 627 - 630 (U.S.S.R.)
Received: 6 / 1957 Reviewed: 7 / 1957

ABSTRACT: Investigations showed that plazothione (in contrast to the statements made by Efros and Levit (ZhOKh, 23, 1552 (1953); 25, 139 (1955)) very energetically associates chlorine as well as bromine while developing that. The reaction is realized in the melting as well as in the medium of organic solvents (chloroform, dioxane, ethane etc.) with or without catalyzers (iron filings). The products of the reaction are tetrachloro- and tetrabromo-tetrahydrobenz-2,3-thiodiazoles which occur in form of a mixture of stereoisomers. On the strength of investigations it can be assumed that on the occasion of interaction between plazothione and chlorine- and bromine an association of 4 halide atoms takes place, on which occasion tetrahalide-tetrahydro-derivatives develop, which, in the case of collaborating with an alcoholic solution of a base, split off two molecules of halide-hydrogen (HCl or HBr) under formation of dihalide derivatives.

Card 1/2

PA - 3.63

Inquiries into the Chemistry of piaz thiole (3,4-benz-1,2,5-thiadiazole).

None of the 4 synthetically obtained isomers correspond to that with the melting point at 87°C described by Efros and Levit. It was found that also 5-methyl- and 6-methyl- and other piazothiole derivatives enter into interacting reaction with chlorine or bromine. It was shown that also 1,2,4,5-tetrahydro-3-thiadiazole enter into such an interacting reaction. The experimental data obtained show that piazthiole (as well as 1,2,4,5-tetrahydro-3-thiadiazole) and its derivatives have properties, beside their aromaticity, which are characteristic for unlimited compounds. A description of experiments follows. (3 citations from Slavic publications).

ASSOCIATION: Leningrad Chemical-Pharmaceutic Institute,
(Leningradskiy khimiko-farmatsevticheskij institut).
PRESENTED BY: NAZAROV, I.N., Member of the Academy
SUBMITTED: 28 July 1956
AVAILABLE: Library of Congress

Card 2/2

PESIN, V.G.; BZhinskii, I.I.; and Kuznetsov, A.I.

2,1,3-Tria- and benzotriazole. Part 3: Halogenation of 2,1,3-benzotriazole and its halo-, mono-, and dimethyl derivatives. Zhur. ob. khim. 34 no.9:3028-3034 3 '64.

MIWA 10:11

1. Leningradskiy khimiko-farmatsevticheskiy institut.

AUTHORS: Khaletskiy, A. N., Pesin, V. G. , 20-114-4-36/63
Chzhou Ts'in'

TITLE: Investigations on the Chemistry of Pyrazothiol (Issledovaniye v oblasti khimii piaztiola) The Oxidation of 3,4-Benzo-1,2,5,-Thiodiazole and Its Derivatives (Okisleniye 3,4-benz-1,2,5-tiodiazola i yego proizvodnykh)

PERIODICAL: Doklady Akademii Nauk SSSR, 1957, Vol. 114, Nr 4, pp. 811-814 (USSR)

ABSTRACT: It has been reported earlier that pyrazothiol and its derivatives, as well as 1',2'-naphthopyrazothiol, are characterized by a certain unsaturatedness besides having aromatic properties. Thus it is not possible to realize, with regard to pyrazothiol, some reactions characteristic for the ethylene compounds (addition of HCl, diene synthesis). It was found that the unsaturatedness of pyrazothiol itself is diminished in its 1,4-dichloro derivative; The aromatic character of this latter compound is however increased. In this connection it was of interest further to investigate the unsaturated properties of pyrazothiol. For this purpose the authors used oxidation. In the

Card 1/3

Investigations on the Chemistry of Pyrazothiol. The Oxidation of 3,4-Benzo-1,2,5-Thiodiazole and Its Derivatives 20-114-4-36/63

saturatedness than pyrazothiol. It also reacts less energetically than pyrazothiol. In the ozonization of pyrazothiol, an ozonide with a melting of 60°C was obtained. After its decomposition a mixture of 2,1,3-thiodiazole-4,5-dicarboxylic acid, 2,1,3-thiodiazole-4,5-dialdehyde, and glyoxal developed. The two latter substances were isolated as the corresponding semi-carbazones; besides that a small amount of ammonium sulfate formed. From the results of the oxidation of pyrazothiol and some of its derivatives may be seen that it is of an unsaturated nature, as it had been found out previously from examples of haloidanion. There are 4 references, 3 of which are Soviet.

ASSOCIATION: Leningrad Chemico-Pharmaceutical Institute (Leningradskiy khimiko-farmatsevticheskiy institut)

PRESENTED: March 12, 1957 by I. N. Nazarov, Member, Academy of Sciences, USSR

SUBMITTED: January 31, 1957

Card 3/3

1. BIN, Y. G., and J. H. G. (1971) "The ...
... 0,1,2-... .."
17 ... (...),
... , ...)

PESIN, V. G.

AUTHORS: Pesin, V. G. Khaletskiy, A. M.

TITLE: Investigations in the Field of the Chemistry of 2,1,3-thio-
diazole (Issledovaniya v oblasti khimii 2,1,3-tio-diazola,
VI. Chlorination and Bromination of 4- and 5-Aminobenz-2,1,3-
-Thiodiazoles (VI. Khlorigirvaniye i bromirovaniye 4- i 5-
aminobenz - 2,1,3 - tiodiazolov)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 1, pp. 2-4 (USSR)

ABSTRACT: The authors earlier showed that 4- and 5-aminobenz-2,1,3-
-thiodiazoles are easily acylated under conditions which are
customary for aromatic amines. The present paper gives data
which were obtained in the investigation of the chlorination
and bromination-reaction of 4- and 5-aminobenz-2,1,3-thio-dia-
zoles. It was shown that in the chlorination of 4-aminobenz-
-2,1,3-thiodiazole with dichloroamine a mono-2,1,3-dichloro-
benz-2,1,3-thiodiazole with a good yield forms; in the bromi-
nation of the same product with bromine in acetic acid (cat.)
no-5,7-dibromobenz-2,1,3-thiodiazole with a good yield (80%).

Card 1/3

Investigation in the Field of the Chemistry of 2,1,3-Thiadiazole-5-thioamides
Bromination and Chlorination of 4- and 5-Aminobenz-2,1,3-Thiadiazole

and more) is obtained. It became evident that the presence of chlorine or bromine upon melting of the diazole 4-amino-5-thioamide and accordingly 5-amino-4-bromobenz-2,1,3-thiadiazole was produced. In the synthesis of 5-amino-4-bromobenz-2,1,3-thiadiazole-5-thioamide (1,3-thiadiazole forms, in the chlorination with 1,3-thioamide-5-amino-6-chlorobenz-2,1,3-thiadiazole. Under the influence of hydrocyanic acid upon 5-amino-4-bromobenz-2,1,3-thiadiazole-5-thioamide and 5-amino-4-chlorobenz-2,1,3-thiadiazole-5-thioamide are produced which are in turn converted to the corresponding thio derivatives. The synthesis of 6-(2-aminothiole)-benz-1,3-thiadiazole is realized by the influence of thiocyanic acid upon 4-bromobenz-2,1,3-thiadiazole as well as by bromination of benz-2,1,3-thiadiazole-5-thioamide. There are 13 references, 15 of which are Slavic.

ASSOCIATION: Leningrad Chemical-Pharmaceutical Institute (Leningrad Institute of Chemical Technology)

Card 2/3

Investigation in the Field of the Chemistry of 2,1,3-Thiadiazole. VI. Chlorination and Bromination of 4- and 5-Aminobenz-2,1,3-Thiadiazoles

2/17/63

SUBMITTED: January 17, 1957

AVAILABLE: Library of Congress

Card 3/3

1. Acylation
2. Chlorination-Reaction
3. Bromine-Reaction
4. Chemistry

PESIN V G

AUTHORS: Pesin, V. G., Knaletskiy, A. I.

7-2-3/1

TITLE: Investigations in the Field of the Chemistry of 2,1,3-thiadiazole
(Issledovaniya v oblasti khimii 2,1,3-thiadiazola)
VII. The Thiocyanogenation of 4- and 5-Aminobenz-2,1,3-thiadiazoles
(VII. Redanirovaniye 4- i 5-aminobenz-2,1,3-thiadiazolov)

PERIODICAL: Zhurnal Obshchey Khimii, 1956, Vol. 26, Nr 2, p. 33-36 (USSR)

ABSTRACT: In the preceding report (reference 1) it was shown that 4-amino-5,7-dihalogenbenz-2,1,3-thiadiazoles are formed by the chlorination and bromination of 4-aminobenz-2,1,3-thiadiazole. By the chlorination and bromination of 5-aminobenz-2,1,3-thiadiazole, however, 5-amino-6-halogenbenz-2,1,3-thiadiazoles form. The thiocyanogenation takes place quite analogously. The thiocyanation was performed according to Kaufman (reference 2) with thiocyanate which formed in the interaction of bromine with ammonium thiocyanate (potassium) in the presence of ammonium bromide (potassium) in the medium of an inert solvent (methanol) at 0 - 2°C. Conclusions: 1) The authors found that 4-amino-5,7-dithiocyanobenz-2,1,3-thiadiazole with a large yield (95 %) forms in the thiocyanation of 4-aminobenz-2,1,3-thiadiazole. On heating with diluted hydrochloric acid it is (probably) converted to 7-thiocyanobenz-2,1,3-(2-

Card 1/3

7-2-73

Investigations in the Field of the Chemistry of 2,1,3-Thiadiazole. VII. The Thiocyanogenation of 4- and 5-Aminobenz-2,1,3-Thiadiazoles

aminothiazole)-benz-2,1,3-thiadiazole. 2) It was found that 5-amino-5-thiocyanogenbenz-2,1,3-thiadiazole with a yield of 40% forms in the thiocyanogenation of 5-aminobenz-2,1,3-thiadiazole. On heating with diluted hydrochloric acid the former is isomerized to 5,6-(2'-aminothiazole)-benz-2,1,3-thiadiazole. 3) It was found that 5-thiocyanogen-4-nitro-1,3-diaminobenzole with an almost quantitative yield forms in the thiocyanogenation of 4-nitro-1,3-diaminobenzene. On heating with diluted hydrochloric acid it isomerizes to 2,5-diamino-5-nitrobenzthiazole. 4) An accessible method for the synthesis of 2,5,6-triaminobenzthiazole with a 10% yield was found. 5) It was shown that 5,6-(2'-aminothiazole)-benz-2,1,3-thiadiazole forms in the interaction of the chlorhydrate of 2,5,6-triaminobenzthiazole with thionylaniline (in the presence of potassium acetate). 6) It was shown that the thiadiazole cycle is under identical conditions considerably easier formed than the imidazole cycle. 7) The reaction of the interaction of thionylaniline with o-diamines of the aromatic series was extended to the o-diamines of the benzthiazole series, which contain an amino group in position 2. Besides it was shown that under certain conditions the diamines can enter into a reaction with thionylaniline, not only in the form of salts, but also as salts. There are 6 references,

Card 2/5

Investigations in the Field of the Chemistry of 2,1,3-Thiodiazole. VII. The Thio-
cyanogenation of 4- and 5-Aminobenz-2,1,3-Thiodiazoles 79-2-23/61

2 of which are Slavic.

ASSOCIATION: **Leningrad Chemical and Pharmaceutical Institute**
(Leningradskiy khimiko-farmatsevticheskiy institut)

SUBMITTED: January 17, 1957

AVAILABLE: Library of Congress

Card 3/3

107,77- 2-3-13, 66
 Resin, V. G., Chelatskiy, L. N., Chzhou, 'Isin'
 Investigations Concerning the Chemistry of the 2,1,3-Thio-
 diazoles (Issledovaniya v oblasti khimii 2,1,3-thiodiazolov)
 VIII. Oxidation of Benzo-2,1,3-Thiodiazole and its Derivatives
 (VIII. Okisleniye benz-2,1,3-thiodiazola i yego proizvodnykh)
 Zhurnal obshchey khimii, 1954, Vol. 29, No. 3, pp. 503-504

Since they were not successful in previous attempts in carrying out some of the reactions characteristic of thio-diazole compounds (addition of HCl, diene synthesis) with benzo-2,1,3-thiodiazole, the authors were interested in further studying the unsaturation properties of this compound and its derivatives in regard to oxidation. In oxidizing the benzo-2,1,3-thiodiazole and its 5-methyl, 5-chloro, and 4,7-dichloro derivatives the organic molecule was decomposed and the sulfur could be recovered only as ammonium sulfate. In the oxidation of 5-chlorobenzo-2,1,3-thiodiazole a small amount of 2,4-dinitrophenylhydrazone (probably as the glycol aldehyde) separated out in addition to the ammonium sulfate.

Investigation Concerning the Chemistry of the 2,1,3-Thiodiazoles.
Oxidation of Benzo-2,1,3-Thiodiazole and Its Derivatives

AY/70-23-6-11,66

In the oxidation of benzo-2,1,3-thiodiazole the oxidation products could be determined only qualitatively. No reaction could be observed when perbenzoic acid was added to this compound at 0°. In the oxidation of benzo-2,1,3-thiodiazole with potassium permanganate, however, two products resulted. One of them probably was an oxide of 2,1,3-thiodiazole-1,2-dicarboxylic acid (Formula 1), as was indicated by its tendency to form double metallic derivatives. The basis of the second showed it to have the composition of 2,1,3-benzo-2,1,3-thiodiazole oxilime with ozone to form ammonium sulfate; 1,2,3-thiodiazole-4,5-dicarboxylic acid; 1,2,3-thiodiazole-4,5-diazoxide; and glyoxalate. There are 3 references, 2 of which are Soviet.

ADDITION: Leningradskiy khimiko-farmatsevticheskiy institut
(Leningrad Chemical-Pharmaceutical Institute)

June 1966

Investigations Concerning the Chemistry of the 2,1,3-Thiodiazole.
VIII. Oxidation of Benzo-2,1,3-Thiodiazole and Its Derivative.

807/79-28-8-1-66

PREPARED: July 15, 1957

Page 10

Author: Resin, V. G., Karlovskiy, A. M., Chukhlov, G. A. *1968-08-01 / 01*

TITLE: Investigations Concerning the Chemistry of the 2,1,3-thio-
 diazoles (Issledovaniya v oblasti khimii 2,1,3-tiolindiazolov)
 IX. The Decomposition Reaction of o-Benzoquinonedioxime With
 Sulfur Dichloride (IX. O vzaimodeystvii o-benzokhinendioksimov
 s dvukhloridom sery)

PERIODICAL: Zhurnal obshchey khimii, 1968, Vol. 38, Nr 8, pp. 1094-1096
 (USSR)

ABSTRACT: The authors undertook the synthesis of compounds which in
 earlier investigations (Refs 1, 2) had shown the properties
 of benzo-2,1,3-thiodiazole (Formula 1) and its derivatives.
 Compounds about whose quinoidal structure there could be no
 doubt were used as starting materials. According to the
 literature (Ref 3) benzofurazan (II) does not react with
 phosphorous pentasulfide, so it was assumed that the acid
 in the benzofuroxan cyclic diazole (III) is less strongly
 bound to the nitrogen atoms than it is in the benzofurazan
 (see the formula diagram). It was thus attempted to synthesize
 benzo-2,1,3-thiodiazole (I) or its oxide (IV) by reacting

Card 1/3

Investigations Concerning the Chemistry of the 2,1,3-Thiadiazoles.
IX. The Decomposition Reaction of o-Benzoquinonediimine with Sulfur Dioxide

NOV 70-22-9-23/66

benzofuroxan (III) with phosphorous pentasulfide or with hydrogen sulfide. This attempt was not successful, nor were the desired results achieved by reacting o-benzoquinonediimine with H₂S in the presence of sodium acetate. In both instances benzofuroxan (II) was produced instead of (i) (a dehydration reaction occurred). These results led to the assumption that with the introduction of compounds which prevent dehydration conditions, i.e. the compounds which prevent dehydration, the formation of the thiadiazole ring is not prevented. Therefore, reactions between o-benzoquinonediimine and thionyl chloride, thionyl aniline, and sulfur dichloride were carried out. These studies showed that thionyl chloride and thionyl aniline do not react with o-benzoquinonediimine. Only a derivative of the benzo-2,1,3-thiadiazole was obtained by the reaction of this oxide with sulfur dichloride. The structure of this derivative is that shown in formula (IV) or (V). There are 5 references, 1 of which is Soviet.

Card 2/3

Investigations Concerning the Disappearance of V. I. ...
NY. The decomposition was to ...

ASSOCIATION: Leningradskiy ...
(Leningrad Chuvstvennoye ...)

DATE: July 15, 1957

KHALETSKIY, A.M.; PESIN, V.G.; CHZHOU TSIN' [Chou Ch'in]

Synthesis and study of N-oxides of heterocyclic compounds. Part 1:
N-oxides of morphine derivatives of tetrahydro isoquinoline and
quinoline. Zhur.ob.khim. 28 no.9:2348-2355 S '58. (MIRA 11:11)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Quinoline) (Isoquinoline) (Morphine)

KHALETSKIY, A.M.; PESIN, V.G.; DEN ZHUN'-SYAN [Teng Jun-hsiang]

Pyrazolidine. Part 1: Synthesis and properties of mono and di substituted 1,2-diphenyl-3,5-dioxypyrazolidine. Zhur.ob.khim. 28 no.9:2355-2359 S '58. (MIRA 11:11)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Pyrazolidine)

AUTHORS: Pesin, V. G., Khaletskiy, A. M.,
Dem'Zhun'-syan SOV 72-08-10-10-00

TITLE: Investigations in the Field of the Chemistry of Pyrazolidine
(Issledovaniya v oblasti khimii pirazolidina) II. Halo-
genation and Thiocyanation of 1,2-Diphenyl-3,5-Dioxo
Pyrazolidine and Its Derivatives (II. Galogenirovaniye
i rodanirovaniye 1,2-difenil-3,5-dioksopirazolidina i yego
proizvodnykh)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 10,
pp 2816 - 2820 (USSR)

ABSTRACT: In contrast to pyrazolones, pyrazolidines which are
structurally closely related to them are insufficiently
investigated. The authors succeeded in easily bringing
1,2-diphenyl-3,5-dioxo pyrazolidine (Compound I) **IB**
into reaction with chlorine, bromine and thiocyanate
under the formation of the corresponding monochloro,
bromo and thiocyano derivatives (II); this was achieved
without catalysts (Scheme 1). The gaseous chlorination
and bromination in the chloroform medium were carried

Card 1/3

Investigations in the Field of the Chemistry of SOV/79-28-10-12 60
Pyrazolidine. II. Halogenation and Thiocyanation of 1,2-Diphenyl-3,5-
Dioxo Pyrazolidine and Its Derivatives

out under cooling. The treatment with thiocyanate formed by the reaction of bromine and thiocyan ammonium in methanol saturated with ammonium bromide was carried out at 0-2°. The corresponding 4-chloro-, 4-bromo-, 4-thiocyano derivatives of 1,2-diphenyl-3,5-dioxo pyrazolidine were separated. It was demonstrated that on the action of a double amount of halogen or thiocyanate (pseudohalogen) on this pyrazolidine its 4,4-dibromo, and 4,4-dithiocyano derivatives (III) and (IV) are formed. The 4,4-dichloro-1,2-diphenyl-3,5-dioxo pyrazolidine could not be obtained. In the chlorination, bromination and thiocyanation of 4-n-butyl-1,2-diphenyl-3,5-dioxo pyrazolidine the corresponding 4-chloro-, 4-bromo, and 4-thiocyano derivatives of 4-n-butyl-1,2-diphenyl-3,5-dioxo pyrazolidine are formed. The 4-bromo-4-n-butyl-1,2-diphenyl-3,5-dioxo pyrazolidine reacts with thiocyanate potassium under the formation of 4-thiocyanate-4-n-butyl-1,2-diphenyl-3,5-dioxo

Card 2/3

Investigations in the Field of the Chemistry of SOV/79-28-10-19,tc
Pyrazolidine. II. Halogenation and Thiocyanation of 1,2-Diphenyl-3,4-
Dioxo Pyrazolidine and Its Derivatives

pyrazolidine (V). There are 4 references, 0 of which
is Soviet.

ASSOCIATION: Leningradskiy khimiko-farmatsevticheskiy institut (Leningrad
Chemical and Pharmaceutical Institute)

SUBMITTED: July 15, 1957

Card 3/3

AUTHORS: Khaletskiy, A. M., Pesin, V. G., Chzhou Tsin' SOV/79-28-10-40/60

TITLE: Synthesis and Investigation of the N-Oxides of Heterocyclic Compounds (Sintez i issledovaniye N-okisey geterotsikliches-kikh soyedineniy) II. N-Oxides of Acridine Derivatives (II. N-Okisi proizvodnykh akridina)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 10, pp 2821-2825 (USSR)

ABSTRACT: Some N-oxides of the acridine series are described. These oxides are of great importance for medicine and are significant as intermediate products of syntheses of all types. The oxidation of the acridine derivatives has been investigated only slightly. The authors investigated the following acridine derivatives: 2-ethoxy-6-nitro-9-chloro acridine (I), 2-ethoxy-6,9-diamino acridine (II), 2-ethoxy-6,9-diacetyl diamino acridine (III), 3,6-diamino acridine (IV), and 3,6-diacetyl diamino acridine (V). In the oxidation of (I) with perbenzoic acid in chloroform the N-oxide (VI) (89% yield) was obtained which was transformed by ammonia in phenol solution into the N-oxide of 2-ethoxy-6-nitro-9-amino acridine (VII); this points to the fact that the

Card 1/3

Synthesis and Investigation of the N-Oxides of Heterocyclic SOV/79-28-10-40/60
Compounds. II. N-Oxides of Acridine Derivatives

N-oxide group does not effect the movability of the chlorine. Perbenzoic acid does not act on (II), whereas peracetic acid causes compound (III) to be formed (18,6% yield) with a subsequent chromatography of the reaction products on aluminum oxide. By the oxidation of this compound with perbenzoic acid the N-oxide (VIII) is formed, which with NaHSO_3 in alcohol diluted with water is transformed into (II). Compound (IV) reacts in a similar way. In the reaction of the two peracids with (IV) only an acylation but no oxidation to the N-oxide is the result. In the oxidation of (V) with perbenzoic acid their N-oxide (IX) is obtained. This proves that the two peracids used cannot be taken for a direct transformation of the amino acridines into the N-oxides. In their oxidation in peracetic acid an acylation first of all takes place and only then the formation of N-oxides of the acyl derivatives. There are 3 references, 1 of which is Soviet.

ASSOCIATION: Leningradskiy khimiko-farmatsevticheskiy institut (Leningrad
Chemical and Pharmaceutical Institute)

Card 2/3

Synthesis and Investigation of the N-Oxides
of Heterocyclic Compounds. II. N-Oxides of Acridine Derivatives

SOV/79-28-10-40/60

SUBMITTED: July 15, 1957

Card 3/3

AUTHORS: Khaletskiy, A. M., Pesin, V. G. SOV/79-28-11-2, 55
Teng Jun-hsiang

TITLE: Investigations in the Field of the Chemistry of 1,2-Di-
pyrazolidine (Issledovaniya v oblasti khimii pirazolidina.
III. Investigation of the Reactivity of 1,2-Diphenyl-3,5-
Dioxypyrazolidine (Nitration, Sulfonation etc.) (III.
Izucheniye reaktsionnoy sposobnosti 1,2-difenil-3,5-
dioksopirazolidina. (Nitrovaniye, sul'firovaniye i dr.))

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 11,
pp 3027 - 3030 (USSR)

ABSTRACT: In the further investigation of the reactivity of
the methylene group of the 1,2-diphenyl-3,5-dioxo-
pyrazolidine (I) its nitration and sulfonation were
carried out. The nitration with the nitration mixture
under cooling yielded the mononitro derivative (II).
The assumption that the nitro group is at the C₄ atom
(II) is concluded from the fact that the hydrogen
at C₄ is highly mobile, and that in the case of the
introduction of the NO₂ group into the benzene nucleus
a dinitro derivative had to be formed. The amino

Card 1/4

Investigations in the Field of the Chemistry of Pyra- SOV/79-28-11-20, 55
zolidine. III. Investigation of the Reactivity of 1,2-Diphenyl-3,5-
Dioxypyrazolidine (Nitration, Sulfonation etc.)

compound formed from it by reduction forms after the diazotization and combination with β -naphthene an unstable dye that could not be separated, as was the case with the 4-aminopyrazole. The sulfonation of the compound (I) after the analysis of the barium salt yields the trisulfo derivative (III), which had to be assumed according to the data given in reference 3, as the latter point to the fact that in the sulfonation of the 1-phenyl-3-methyl pyrazolone-5 (IV) which is similar in structure, the compounds (V), (VI) and (VII) are obtained; the latter have the sulfo groups at the C₄ in the pyrazolone nucleus and in the para-position in the benzene nuclei. In the further reactions of the mentioned sulfo acids with nitrous acid and aryl diazonium compounds the structures (VIII) and (IX) could be attributed to the barium salts of the sulfo acid (VI) and the disulfo acid (VII). In checking these data (Ref 2) with respect to the

Card 2/4

Investigations in the Field of the Chemistry of Pyrazolidine. III. Investigation of the Reactivity of 1,2-Diphenyl-3,5-Dioxypyrazolidine (Nitration, Sulfonation etc.)

sulfonation products of the 1,2-diphenyl-3,5-dioxypyrazolidine the observations made were very similar, so that the structure (II') could be ascribed to it. Thus, the pyrazolidine cycle in the nitration and sulfonation behaves in much the same way as the pyrazolone cycle. Compound (I) easily reacts with n-nitroso-dimethyl aniline and phenyl diazonium chloride according to the mentioned reaction scheme. This fact points to the reactivity of the methylene group. There are 2 Soviet references.

ASSOCIATION: Leningradskiy Khimiko-Farmatsevticheskiy Institut (Leningrad Chemopharmaceutic Institute)

SUBMITTED: July 1958

Card 3/4

AUTHORS: Pepin, V. G., Khaletskiy, A. M., SOV/76-28-11-21, 51
Teng Jun-hsiang

TITLE: Investigations in the Field of the Chemistry of Pyrazolidine (Issledovaniya v oblasti khimii pirazolidina) IV. Synthesis and Investigation of the Sulfides and Disulfides of the 1,2-Diphenyl and 4-n-Butyl-1,2-Diphenyl-3,5-Dioxypyrazolidines (IV. Sintez i issledovaniye sulfidov i disulfidov 1,2-difenil-i 4-n.-butil-1,2-difenil-3,5-dioxypyrazolidinov)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 29, Nr 11, pp 3030 - 3036 (USSR)

ABSTRACT: In the present paper the results of the investigations of the reactivity of chloro, bromo, and thiocyanate derivatives of the 1,2-diphenyl and 4-n-butyl-1,2-diphenyl-3,5-dioxypyrazolidine are described. The 4-bromo-1,2-diphenyl-3,5-dioxypyrazolidine (I) in the reaction with sodium sulfide leads to the formation of the sulfide (I) as main product, and to the compound (III) as secondary product. The

Card 1/3

Investigations in the Field of the Chemistry of
Pyrazolidine. IV. Synthesis and Investigation of the Sulfides and
Disulfides of the 1,2-Diphenyl and 4-n.-Butyl-1,2-Diphenyl-3,5-
Dioxypyrazolidines

SOV, 73-28-11-29/53

4-bromo-4-n.-butyl-1,2-diphenyl-3,5-dioxypyrazolidine (VI) reacts with sodium sulfide under the formation of the sulfide (VII), as well as with triethyl ammonium diethyl phosphate (IV) under the formation of compound (VIII) and (IX), i.e. of thione and thiol structure. It was found that the 4-thiocyano-1,2-diphenyl-3,5-dioxypyrazolidine (X) easily enters reaction with alcoholic alkali liquor and yields the disulfide (XI). In the reaction of the 4-thiocyano-4-n.-butyl-1,2-diphenyl-3,5-dioxypyrazolidine with alcoholate a product without sulfur is obtained as final product. 4-bromo-4-n.-butyl-1,2-diphenyl-3,5-dioxypyrazolidin (VI) with sodium disulfide yields the disulfide of the di-4-n.-butyl-1,2-diphenyl-3,5-dioxypyrazolidine (XII). In the reaction of the bis-malonic ester with hydrazo benzene the bis-1,2-diphenyl-3,5-dioxypyrazolidine (III) (structural proof of the

Card 2/3

Investigations in the Field of the Chemistry of Pyrazolidine. IV. Synthesis and Investigation of the Sulfides and Disulfides of the 1,2-Diphenyl and 4-n.-Butyl-1,2-Diphenyl-3,5-Dioxypyrazolidines SOV/79-28-11-29/55

compound (III) by the other synthesis according to scheme 1) is formed. The experimental results show that in the series of the reactions the n.-butyl group at the C₄ atom exerts a mainly hampering influence on the reaction process. There are 5 references, 4 of which are Soviet.

ASSOCIATION: Leningradskiy Khimiko-farmatsevticheskiy institut
(Leningrad **Chemopharmaceutic** Institute)

SUBMITTED: July 19, 1955

Card 3/3

PESIN, V.G.; HELEN'KAYA-LOTSMANENKO, I.A.; KHALETSKIY, A.M.

2,1,3-Thia- and selenadiazole. Part 34: Alkylation, acylation,
nitration, nitrosation of hydroxy derivatives of benz-2,1,3-
thiadiazole. Zhur. ob. khim. 34 no.11:3763-3768 N '64
(MIRA 18:1)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

AUTHORS: Pesin, V. G., Khaibskiy, A. M., SOV/79-28-12-25/41
Gal'perin, I. S.

TITLE: Investigations in the Field of Pyrazolidine Chemistry
(Issledovaniya v oblasti khimii pirazolidina) V. On Some
Anomalous Reactions of 1,2-Diphenyl-3,5-Dioxo Pyrazolidine
and Its Halogen and Thiocyanate Derivatives (V. O nekotorykh
anomal'nykh reaktsiyakh 1,2-difenil-3,5-dioksopirazolidina i
yego galogen- i rodanproizvodnykh)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 12, pp 3274-3277
(USSR)

ABSTRACT: 1,2-diphenyl- and 4-n.-butyl-1,2-diphenyl-3,5-dioxo pyrazolidine
sometimes react in an anomalous way. The authors showed
already earlier (Ref 1) that in the reaction of 4-n.-butyl-4-
thiocyano-1,2-diphenyl-3,5-dioxo pyrazolidine with alcoholic
alkali a sulfur-free product, but no disulfide is formed. In
this paper some anomalous reactions of 1,2-diphenyl-3,5-dioxo
pyrazolidine and of its 4-bromo derivative are mentioned.
4-bromo-1,2-diphenyl-3,5-dioxo pyrazolidine (I) reacts, for
instance, with potassium thiocyanate under the formation of
compound (II), and not, as expected, of 4-thiocyano-1,2-diphenyl-
3,5-dioxo pyrazolidine. Compound (II) is formed as by-product

Card 1/3

Investigations in the Field of Pyrazolidine Chemistry. SOV/79-28-12-25/41
V. On Some Anomalous Reactions of 4,2-Diphenyl-3,5-Dioxo Pyrazolidine and Its Halogen and Thiocyanate Derivatives

According to reference 1, besides the main product (III), in the reaction of compound (I) with sodium sulfide, or with diethyl thiophosphate of triethyl ammonium (Scheme 1). Based on these reactions it may be assumed that the formation of compound (II) takes place radical-like. Thus, a number of anomalous reactions of 4-bromo-1,2-diphenyl-3,5-dioxo pyrazolidine with potassium thiocyanate, pyridine, p-chlorophenol and others were described; in this reaction products are formed which do not contain any sulfur or halogen. In the reaction of methylene-bis-malonic ester with hydrazo benzene the methylene-bis-4,2-diphenyl-3,5-dioxo pyrazolidine is formed. The anomalies mentioned corresponded probably to the irregular reaction of bromo malonyl chloride with excess hydrazo benzene (Scheme 3) as mentioned in publications (Ref 2). There are 4 references, 2 of which are Soviet.

ASSOCIATION
Card 2/3

Leningradskiy khimiko-farmatsevticheskiy institut (Leningrad
Chem-Pharmaceutical Institute)

PESIN, V.G.; KHALETSKIY, A.M.; KAUKOVA, L.A.

Chemistry of 2,1,3-thiodiazole. Part 11: Chlorination and bromination of 1',2'-naphtho-2,1,3-thiodiazole. Zhur.ob. khim. 30 no.7:2187-2192 J1 '60. (MIRA 13:7)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Thiadiazole)

PESIN, V.G.; SERGEYEV, V.A.; KHALETSKIY, A.M.

2,1,3-Thia- and selenadiazole. Part 32: Behavior of
benz-2,1,3-thiadiazole and its derivatives toward chromic
acid. Zhur. ob. khim. 34 no.11:3753-3756 N '64 (MIRA 18:1)

1. Leningradskiy khimiko farmatsevticheskiy institut.

PESIN, V.G.; D'YACHENKO, S.A.; KHALETSKIY, A.M.

2,1,3-Thia- and selenadiazole. Part 33: di(β -chloroethyl) amino
derivatives. Zhur. ob. khim. 34 no.11:3757-3763 N '64
(MIRA 18:1)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

1965-1970

1965-1970

1965-1970

1. M. G. Kuznetsov, V. I. Kuznetsov, L. A. Gerasimova, *Chemical Pharmaceutical Institute*
Abstracts of the USSR Academy of Sciences, 1965, No. 10, p. 1271.
Reaction of Salts of Diethylthio and Diethylphosphoric Acids. VIII.
Reaction of Salts of Diethylthio and Diethylphosphoric Acids with Aromatic and
Heterocyclic Halo-Derivatives.

Moscow, *Khimicheskoye Sostoyaniye*, Vol. 10, No. 10, 1965, pp 1268-1271.

Abstract: Thiols, disulfides, and thio phosphates, halo-phenyl organic compound
Abstract: In the reaction of potassium diethylthiophosphate with 2,4-
dinitrofluorobenzene, diethylthiophosphate and 2,4-dinitro-thiophenolate
were formed. A mechanism of their formation was proposed. Potassium
diethylthiophosphate was found to react analogously with 2,4-dinitro-
chlorobenzene and 2,4-dinitrofluorobenzene. Potassium diethylthiophosphate
reacted with picryl chloride, 2-chlorobenzothiazole, 2-chloro-5-nitrobenzothia-
zole, 4-chloro-7-nitrobenz-1,2,3-thiadiazole, 2-chloro-5-nitropyridine, and
2-chloro-3,5-dinitropyridine, forming sulfides R-S-R. o-Nitrochlorobenzene,
p-nitrochlorobenzene, 2-bromopyridine, and 8-chlorocaffeine do not take part
in this reaction with potassium diethylthiophosphate. Potassium diethylthio-
phosphate reacted with benzoyl chloride and p-nitrobenzoyl chloride to
form the corresponding thiol derivatives: O,O-diethyl-S-benzoyl thiophosphate
and O,O-diethyl-S-p-nitrobenzoyl thiophosphate, respectively. Orig. art. has:
2 figures and 1 table. [CPHS: 38,970]

SUB CODE: 07 / SUBM DATE: 15Jun65 / ORIG REF: 015 / OTH REF: 024

Card 1/1 30

UDC: 547.26'118
07235-2006

PESIN, V.G.; VITENBERG, I.G.

Reaction of aromatic and heterocyclic thiocyno derivative with
diethyl and triethyl phosphite. Zhur. ob. khim. 35 no.5:930
My '65. (MIRA 18:6)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

PESIN, V.G.; D'YACHIZKO, Ye.K.

Chloromethylation of 2,1,3-benzothiadiazole and its derivatives.
Zhur. ob. khim. 34 no.7:2475 J1 '64 (MIRA 17:8)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

L 17957-65 EWT(m)/EPF(c)/EWP(j) PC-4/Pr-4 RM
ACCESSION NR: AF5002623 S/0079/64/034/008/2769/2773

AUTHOR: Pesin, V. G.; Vitenberg, I. G.; Khaletskiy, A. M. 6

TITLE: Salts of dialkyl- and diarylmonothio- and dithiophosphoric acids. VIII.
Interaction of salts of dialkyl- and diphenyl-dithiophosphoric acids with aromatic
and heterocyclic halo derivatives

SOURCE: Zhurnal obshchey khimii, v. 34, no. 8, 1964, 2769-2773

TOPIC TAGS: phosphoric acid, sulfide, halogenated organic compound, ester,
ammonium salt, elemental halogen

Abstract: Halogen derivatives that form chiefly sulfides with alkali salts
of dialkyl- and diphenylthiophosphoric acids also react with alkali salts
of diphenyldithiophosphoric acid, to form sulfides with the structure R-S-R;
with salts of dialkyldithiophosphoric acids, the corresponding esters of
dithiophosphoric acids, with the structure RSP(S)(OAlk)₂ are principally
formed. The nature of the aromatic and heterocyclic halogen derivatives
and that of the solvent influence the course of the reaction: 2,4-dinitroch-
lorobenzene reacts with the potassium or ammonium salt of diethyldithiophos-
phoric acid in alcohol to form a mixture of 2,4,2',4'-tetranitrodiphenyl
sulfide and 2,4,2',4'-tetranitrodiphenyl disulfide; in acetone, 40% of the

Card 1/2

L 17957-65

ACCESSION NR: AP5002623

sulfide, 2-3% of the disulfide, and some 0,0-diethyl-S-2,4-dinitrophenyl dithiophosphate were formed. When the reaction is conducted with 4-nitro-5-bromobenz-2,1,3-thiadiazole, both in alcohol and in acetone, a mixture of the sulfide and the dithiophosphoric acid ester is formed. The reaction was also studied with the chloro derivatives: 2,4-dinitrochloronaphthalene, 2-ethoxy-5-nitro-9-chloroacridine, 4-bromo-4-butyl-1,2-diphenyl-3-pyrazolidine, and 4-bromantipyrine. Orig. art. has 1 formula and 2 tables.

ASSOCIATION: Leningradskiy khimiko-farmatsevticheskiy institute (Leningrad Chemicopharmaceutical Institute)

SUBMITTED: 19Jun63

ENCL: 00

SUB CODE: OC, GC

NO REF SOV: 007

OTHER: 005

JPRS

Card 2/2

BALONOVA, E.M.; KHALETSKIY, A.M.; PESIN, V.G.

Esters of β -sitosterol. Zhur. ob. khim. 34 no.7:2157-2158
J1 '64 (MIRA 17:8)

1. Leningradskiy khimiko-farmatseticheskiy institut.

PESIN, V. G.; SERGEYEV, V. A.; KHALETSKIY, A. M.

2,1,3-Thia- and selenadiazoles. Part 30: Nitration of mono-
and dimethyl derivatives of benzo-2,1,3-thiadiazole. Zhur.
ob. Khim. 34 no.6:1986-1992 Je '64. (MIRA 17:7)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

PESIN, V.G.; ZOLOTOVA-ZOLOTUKHINA, L.V.; KHALETSKIY, A.M.

2,1,3,-Thiadiazoles and selenadiazole. Part 24: Synthesis and study of
2-mercapto[3,4-e]thiazolo- and [4,5-e]benzo-2',1',3'-thiadiazoles.
Zhur.ob.khim. 34 no.1:255-260 Ja '64. (MIRA 17:3)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

PESIN, V.G.; KHALETSKIY, A.M.; SERGEYEV, V.A.

2,1,3-Triadiazoles and selenadiazoles. Part 25: Direct amination of
2,1,3-benzothiadiazole derivatives. Zhur.ob.khim. 34 no.1:261-272
Ja '64. (MIRA 17:3)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

ZONIS, L.S.; KHALETSKIY, A.M.; PESIN, V.G.

Synthesis and study of 1-[p- β -diethylaminoethoxyphenyl]-p-
tolyl-2-p-chlorophenyl] ethanol. Zhur.ob.khim. 33 no.10:3141-
3142 0 '63. (MIRA 16:11)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

PESIN, V.G.; KHALETSKIY, A.M.; SERGEYEV, V.A.

2,1,3-Thiodiazole. Part 22: Nitration of derivatives of
benzo-2,1,3-thiodiazole. Zhur.ob.khim. 33 no.6:1759-1766 Je
'63. (MIRA 16:7)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Benzothiadiazole) (Nitration)

PESIN, V.G.; KHALETSKIY, A.M.; LOTSMANENKO, I.A.

2,1,3-Thiodiazole. Part 21: Chlorination, bromination, and
nitration of 4- and 5-hydroxybenzo-2,1,3-thiodiazoles. (MIRA 10:7)
Zhur.ob.khim. 33 no.6:1752-1759 Je '63.

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Benzothiadiazole) (Halogenation) (Nitration)

PESIN, V.G.; KHALETSKIY, A.M.; LOTSMANENKO, I.A.

2,1,3-Thiodiazole. Part 20: Sucherer reactions and diazotization involving amino derivatives of benzo-2,1,3-thiodiazole. Zhur.ob.khim. 33 no.6:1746-1752 Je '63. (MIRA 16:7)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Benzothiazole) (Amino compounds)

PESIN, V.G.; KHALETSKIY, A.M.; LOTSMANENKO, I.A.

Chemistry of 2,1,3-thiodiazole. Part 18: Esters and amides of
5,7-dihalobenzo-2,1,3-thiodiazole-4-hydroxyacetic acids. Zhur. obshch.
khim. 33 no.4:1096-1101 Ap '63. (MIRA 16:5)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Thiadiazole) (Acetic acid)

PESIN, V.G.; KHALETSKIY, A.M.; ZOLOTOVA-ZOLOFURHINA, L.V.

Chemistry of 2,1,3-thio- and selenodiazoles. Part 19:
Synthesis of 2-methylthiazolo (5,4-g)- and 2-methylthiazolo
(4,5-g) benzo-2', 1',3'-thiodiazoles and their seleno analogs,
Zhur.ob.khim. 33 no.4:1101-1104 Ap '63. (MIRA 16:4)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Thiazole) (Thiadiazole) (Selenium organic compounds)

PESIN, V.G.; KHALETSKIY, A.M.; SERGEYEV, V.A.

Chemistry of 2,1,3-benzothiadiazole. Part 17: Halogenation
of 2,1,3-benzothiadiazole and its halo derivatives. Zhur.ob.khim.
33 no.3:949-952 Mr '63. (MIRA 16:3)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Benzothiadiazole) (Halogenation)

PESIN, V.G.; KHALETSKII, A.M.; VITENBERG, I.O.

Salts of dialkyl phosphorothioic acids. Part 5: Interaction
of salts of dialkyl phosphorothioic acids with aromatic
halogen derivatives. Zhur.ob.khim. 33 no.2:388-391 F '63.
(MIRA 16:2)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Phosphorothioic acid) (Halogen compounds)

PESIN, V. G.; KHALETSKIY, A. M.; SERGEYEV, V. A.

Chemistry of 2,1,3-thiadiazole. Part 16: Chlorination,
bromination and thiocyanation of 5-aminobenzo-2,1,3-
thiadiazole. Zhur. ob. khim. 33 no.1:230-233 '63.
(MIRA 16:1)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

(Thiadiazole)

PESIN, V.G.; KHALETSKIY, A.M.; D'YACHENKO, Ye.K.

Chemistry of 2,1,3-thiodiazole. Part 15: Oxidation
of benzo-2,1,3-thiodiazole by potassium permanganate.
Zhur.ob.khim. 32 no.11:3505-3510 N '62. (MIRA 15:11)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Thiadiazole) (Oxidation)

PESIN, V.G.; KHALETSKIY, A.M.

Chemistry of 2,1,3-thio and selenodiazoles. Part 14:
Reactivity of bromine in derivatives of 2,1,3-benzothiadiazole.
Zhur.ob.khim. 32 no.10:3284-3290 0 '62. (MIRA 15:11)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Benzothiadiazole) (Bromine)

KHALETSKIY, A.M.; PESIN, V.G.; VITENBERG, I.G.

Synthesis of amides of β -phenylisopropylamine and of some carboxylic acids. Zhur.ob.khim. 32 no.4:1068-1071 Ap '62. (MIRA 15:4)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Isopropylamine) (Amides)

PESIN, V.G.; KHALETSKIY, A.M.; ZOLOTOVA-ZOLOTUKHINA, L.V.

Chemistry of 2,1,3-thio- and selendiazole. Part 12: Synthesis and study of derivatives of pyrimidine-2,1,3-thio and selendiazole.
Zhur.ob.khim. 31 no.9:3000-3003 S '61. (MIRA 14:9)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Pyrimidine) (Selenium organic compounds)

ZONIS, L.S.; KHALETSKIY, A.M.; PESIN, V.G.

Synthesis and study of some 5,5'-dialkylaminoalkyl derivatives of
barbituric and thiobarbituric acids. Zhur.ob.khim. 31 (MIRA 14:9)
no.9:3004-3006 S '61.

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Barbituric acid)

PESIN, V.G.; KHALETSKIY, A.M.

Salts of dialkylthiophosphoric acids. Zhur. ob khim. 31 no.8:2508-
2515 Ag '61. (MIRA 14:8)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Phosphorothioic acid)

PESIN, V.G.; KHALETSKIY, A.M.

Salts of dialkylthiophosphoric acids. Part 2: Properties of
di-(β -chloroethyl)-phosphorous acid. Zhur. ob. khim. 31 no.8:
2515-2518 Ag '61. (MIRA 14:8)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Phosphorous acid)

PESIN, V.G.; KHALETSKIY, A.M.

Salts of dialkylthiophosphoric acids. Part 3: **Reactivity**
and structure of diethylthiophosphoric acid salts. Zhur.
ob. khim. 31 no.8:2518-2522 Ag '61. (MIRA 14:8)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Phosphorothioic acid)

PESIN, V.G.; KHALETSKIY, A.M.; VITENBERG, I.G.

Salts of dialkylthiophosphoric acids. Part 4: Reactions of
dialkylthiophosphoric acid salts with aromatic and heterocyclic
halogen derivatives. Zhur. ob. khim. 31 no.8:2522-2526 Ag '61.
(MIRA 14:8)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Phosphorothioic acid) (Halogen compounds)

PESIN, V.G.; KHALETSKIY, A.M.; SERGEYEV, V.A.

2,1,3-Thio- and selenodiazoles. Part 13: Nitration of 4- and 5-aminobenz-2,1,3-thiodiazoles. Zhur. ob. khim. 32 no.1:181-186 Ja '62. (MIRA 15:2)

1. Leningradskiy khimiko-farmatsevticheskiy institut.
(Thiadiazole) (Nitration)

PESIN, V.G.; VITENBERG, I.G.; KHALETSKIY, A.M.

Salts of dialkyl- and diarylphosphorodithioic and dithio-
phosphorodithioic acids. Part 7: Interaction of the salts
of dialkyl- and diphenylphosphorodithioic acids with aromatic
and heterocyclic male derivatives. Zhur. ob. khim. 34 no.8:
2769-2773 A; 104. (MIRA 17:9)

1. Leningradskiy khimiko-farmatsevticheskiy institut.

ACCESSION NR: AP5011028

UR/0079/64/034/011/3753/3756

AUTHOR: Pesin, V. G.; Sergeyev, V. A.; Khaletskiy, A. M. 1
B

TITLE: Investigations in the field of 2, 1, 3-thia- and -selenadiazole. XXXII. Behavior of benz-2, 1, 3-thiadiazole, and its derivatives toward chromic acid

SOURCE: Zhurnal obshchey khimii, v. 34, no. 11, 1964, 3753-3756

TOPIC TAGS: chromic acid, organic nitrogen compound, organic azo compound, organic sulfur compound

Abstract: Under the action of chromic anhydride in sulfuric acid on benz-2,1,3-thiadiazole, its 4- and 5-methyl- and 5,6-dimethyl derivatives, 2,1,3-thiadiazole-4,5-dicarboxylic acid was formed in satisfactory yield. In the case of 5-bromobenz-2,1,3-thiadiazole, in addition to the latter, 4,5-dibromobenz-2,1,3-thiadiazole was formed. 5-Chloro-, 4,7-dichloro-, 4,7-dibromo-, 4-nitro-, and 5-nitrobenz-2,1,3-thiadiazoles manifested high stability toward this oxidizing agent. In the reaction of 2,1,3-thiadiazole-4,5-dicarboxylic acids with organic bases (aniline, lower aromatic toluidines, pyridine, p-anisidine, p-phenetidine, and piperazine, the corresponding acid salts were produced; with beta-phenyl-isopropylamine, dimethylaminoethylamine, and piperidine, neutral salts were formed. Orig. art. has 7 formulas and 3 tables.

Card 1/2

ACCESSION NR: AP5011028

ASSOCIATION: Leningradskiy khimiko-farmatsevticheskiy institut (Leningrad
Chemicopharmaceutical Institute)

SUBMITTED: 26Jun63

ENCL: 00

SUB CODE: OC, GC

NO REF SOV: 010

OTHER: 015

JPRS

Card 2/2

ACCESSION NR: AP5011029

UR/0079/64/034/011/3757/3762

AUTHOR: Pesin, V. G.; D'yachenko, S. A.; Khalitskiy, A. N. 1
B

TITLE: Investigation in the field of 2,1,3-thia- and -selenadiazole. XXXIII. Di (beta-chloroethyl) amino derivatives

SOURCE: Zhurnal obshchey khimii, v. 34, no. 11, 1964, 3757-3762

TOPIC TAGS: organic azo compound, organic nitrogen compound, organic sulfur compound

Abstract: 4- and 5-Aminobenz-2,1,3-thiadiazoles were found to react with ethylene oxide, forming 4- and 5-beta-hydroxyethyl)aminobenz-2,1,3-thiadiazoles. The reaction of 5-methyl-4-amino, 4-methyl-5-amino-, and 4-methyl-7-aminobenz-2,1,3-thiadiazoles with ethylene oxide produced the corresponding di(beta-hydroxyethyl)amino derivatives of benz-2,1,3-thiadiazole. 5-Methyl-4-amino-benz-2,1,3-selenadiazole formed 5-methyl-4-di(beta-hydroxyethyl)aminobenz-2,1,3-selenadiazole. The reaction of 4- and 5-bromoethylbenz-2,1,3-thiadiazoles, 5-bromo-4-nitro- and 5-methoxy-4-nitrobenz-2,1,3-thiadiazoles with diethanolamine produced the corresponding di(beta-hydroxyethyl)amino derivatives of benz-2,1,3-thiadiazole. 5-Bromo-4-nitrobenz-2,1,3-selenadiazole formed 4-nitro-5-

Card 1/2

ACCESSION NR: AP5011029

(beta-hydroxyethyl)aminobenz-2,1,3-selenadiazole in the reaction with diethanolamine. The reaction of the di(beta-hydroxyethyl)-amino derivatives of benz-2,1,3-thia- and -selenadiazoles with thionyl chloride produced the corresponding di(beta-chloroethyl)amino derivatives of benz-2,1,3-thia- and -selenadiazoles. Orig. art. has 1 table.

ASSOCIATION: Leningradskiy khimiko-farmatsevticheskiy institut (Leningrad
Chemico-pharmaceutical Institute)

SUBMITTED: 26Jun63

ENCL: 00

SUB CODE: OC, GC

NO REF SOV: 006

OTHER: 003

JPRS

Card 2/2

ACCESSION NR: AP5011030

UR/00179/64/034/011/3763/3768

AUTHOR: Pesin, V. G.; Belen'kaya-Lotsmanenko, I. A.; Khaletskiy, A. M. 1
B

TITLE: Investigations in the field of 2,1,3-thia- and -selenadiazole. XXIV. Alkylation, acylation, nitration, and nitrosylation of hydroxy derivatives of benz-2, 1, 3-thiadiazole

SOURCE: Zhurnal obshchey khimii, v. 34, no. 11, 1964, 3763-3768

TOPIC TAGS: organic azo compound, organic nitrogen compound, alkylation, nitration, nitric acid, organic sulfur compound

Abstract: Alkylation and acylation of 4- and 5-hydroxybenz-2,1,3-thiadiazoles produced 4- and 5-methoxy-, -ethoxy, and -acetoxybenz-2,1,3-thiadiazoles, respectively. In the nitration of 4- and 5-ethoxybenz-2,1,3-thiadiazoles, 4-ethoxy-5,7-dinitro- and 5-ethoxy-4-nitrobenz-2,1,3-thiadiazoles were produced, respectively. Nitration of 4-hydroxy-7-nitrobenz-2,1,3-thiadiazole, as well as hydrolysis of 4-chloro-5,7-dinitro- and 4-ethoxy-5,7-dinitrobenz-2,1,3-thiadiazole produced 4-hydroxy-5,7-dinitrobenz-2,1,3-thiadiazole; the latter was reduced with sodium hyposulfite to 4-hydroxy-5,7-diaminobenz-2,1,3-thiadiazole. In the nitrosylation of 4- and 5-hydroxybenz-2,1,3-thiadiazoles, 4-hydroxy-7-nitroso- and 5-hydroxy-4-nitrosobenz-2,1,3-thiadiazoles were

Card 1/2

ACCESSION NR: AP5011030

obtained, respectively. 4-Hydroxy-7-amino- and 5-hydroxy-4-aminobenz-2,1,3-thiadiazoles, were produced by reducing the corresponding nitro- and nitroso-derivatives. 4-Hydroxy-7-aminobenz-2,1,3-thiadiazole readily formed 4-hydroxy-7-acetaminobenz-2,1,3-thiadiazole when treated with acetic anhydride. Treatment of the product with ethyl iodide resulted in the formation of a black mass, from which 4-ethoxy-7-acetaminobenz-2,1,3-thiadiazole could not be isolated. A study of the methylation of 4- and 5-hydroxybenz-2,1,3-thiadiazoles according to Friedel-Crafts showed that the reaction does not take place under normal conditions, evidently as a result of the deactivating influence of the thiadiazole ring. Oxazole /4,5-e/benz-2,1,3-thiadiazole was produced by the action of 5-hydroxy-4-aminobenz-2,1,3-thiadiazole with orthoformic ester.

Orig. art. has 2 formulas.

ASSOCIATION: Leningradskiy khimiko-farmatsevticheskiy institut (Leningrad Chemical-pharmaceutical Institute)

SUBMITTED: 11Jul63

ENCL: 00

SUB CODE: OC, GC

NO REF SOV: 010

OTHER: 001

JPBS

Card 2/2

PERMITS AND PROPERTIES INDEX

A-1-8

BC

Manufacture of titanium dioxide from sphero-
 granules. V. M. Tsvetkovskaya, Y. M. [?], A. L.
 Kostin, and S. S. Frenkel (J. Appl. Chem., Russia,
 1952, 5, 688-689) (containing in Russian) with 80%
 H₂SO₄ at 140-150° for 2-3 hr. The dry, powdered
 residue is extracted with H₂O in 20% H₂SO₄ and the
 solution reduced with Fe and hydrolyzed, the product
 being washed and ignited at 800° for 3 hr. The yield
 of TiO₂ is 91%. (Chem. Abstr. 47:1000)

METALLURGICAL LITERATURE CLASSIFICATION

E-2

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
---	---	---	---	---	---	---	---	---	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	-----

PROCESSES AND PROPERTIES INDEX

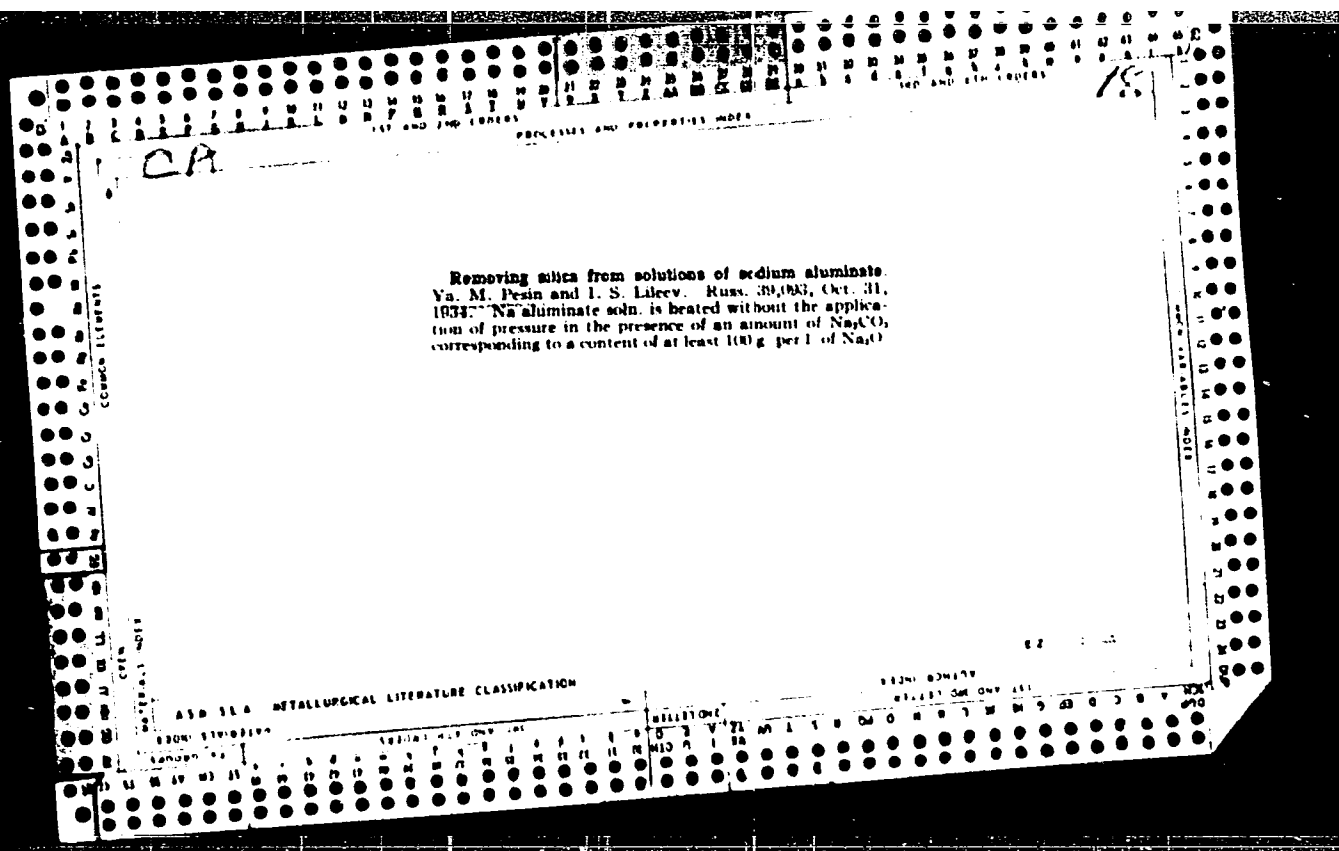
Manufacture of titanium dioxide from sphene-rutile. V. E. TISHCHENKO, YA. I. M. PRIN, A. L. KOSIRAK AND S. S. FRIEDLICH. *J. Applied Chem. (U. S. S. R.)* 5, 685-688 (1952). Content of the ore is required. Titanite is digested with 80% H₂SO₄ (1 part concentrate/1.5 parts actual H₂SO₄) at 140-5° for 10-11 hrs. The dry and powder (1 part concentrate/1.5 parts actual H₂SO₄) at 140-5° for 7 hrs. This soln. is reduced with Fe and hydrochloric is extd. with water at 0° for 7 hrs. The yield of TiO₂ is 71% lyzed. The product is washed and ignited at 500° for 3 hrs. The yield of TiO₂ is 71% lyzed. V. KALICHIRVAY

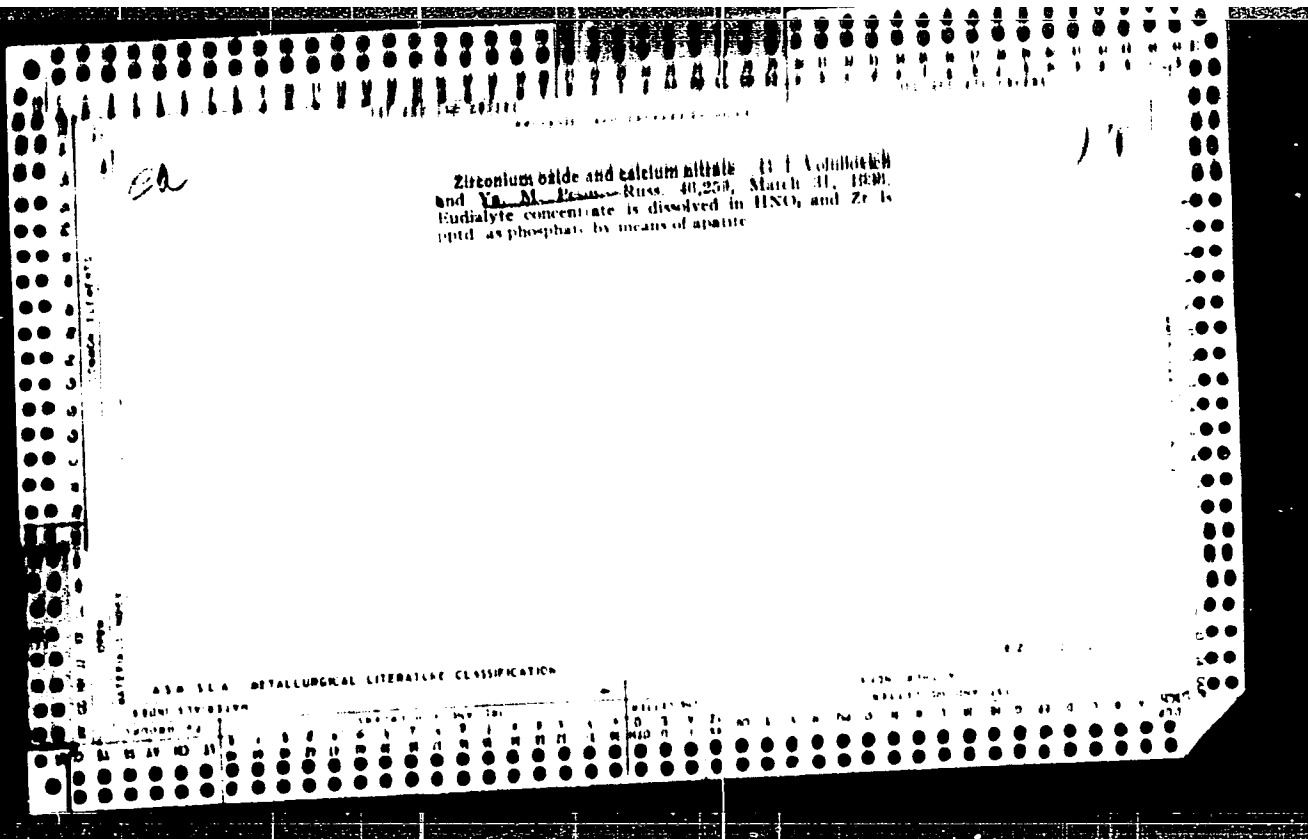
ASB-514 METALLURGICAL LITERATURE CLASSIFICATION

ZUBOV, V.Ya.; GRACHEV, S.V.; PESIN, Ya.A.

Stress relaxation during martensite transformations of residual austenite. Izv.vys.ucheb.zav.; Chern.Met. no.5:115-120 '60. (MIRA 13:6)

1. Ural'skiy politekhnicheskiy institut.
(Steel--Metallography) (Strains and stresses)





197 AND 198 ORDERS

PROCESSES AND PROPERTIES INDEX

78

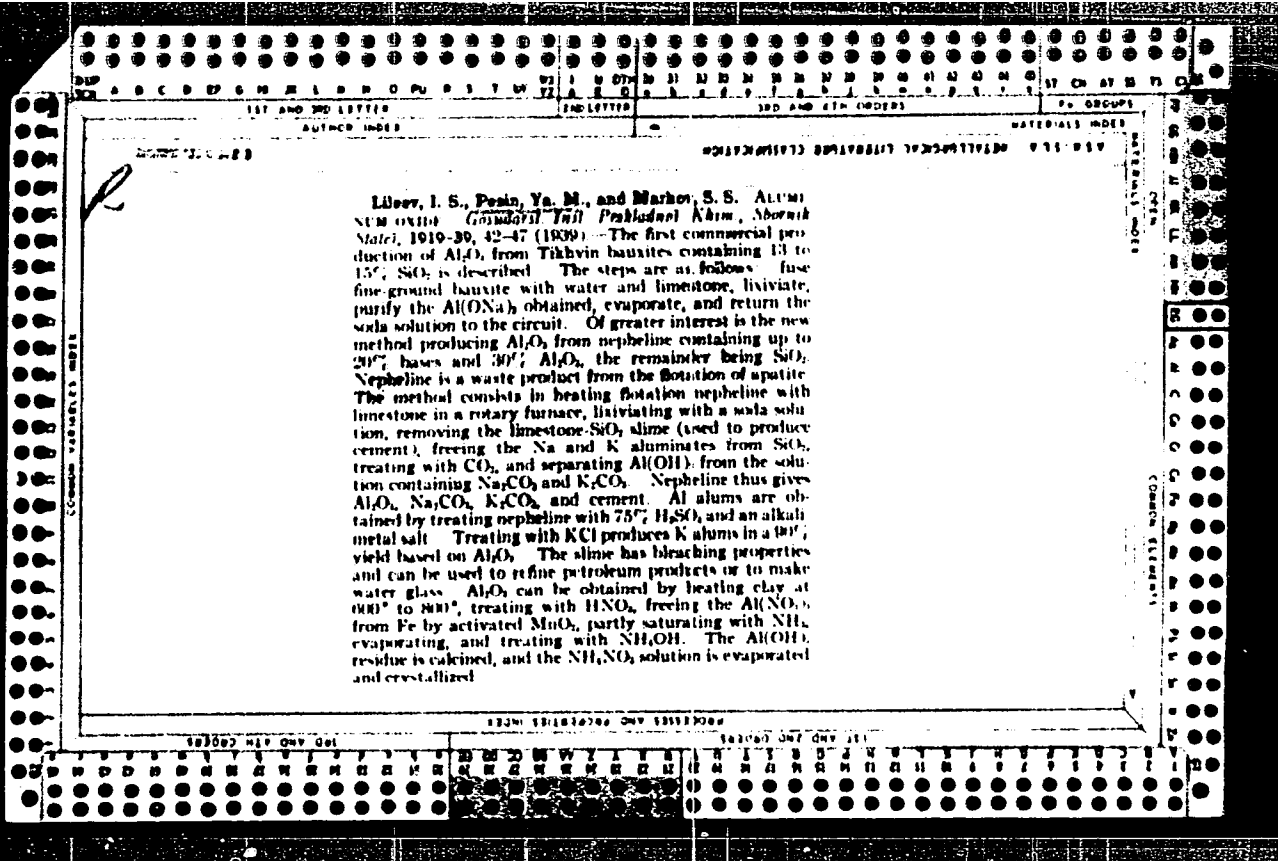
Con

Zirconium hydroxide Ya. M. Persa. Russ. 22,075, Oct. 31, 1937. Acidic solns. of Zr salts contg. also salts of Fe, Al, etc., are partially neutralized at boiling temp. to the extent that the ratio of acid to total oxide is 0.85 2 0. In this range, Zr(OH)₂ pptn. with a small amt. of Fe; the other metals remain in soln.

ASD-51.6 METALLURGICAL LITERATURE CLASSIFICATION

197 AND 198 ORDERS

197 AND 198 ORDERS



PRECIPITATION AND PREPARATION METHODS

18

cd

Aluminum oxide. I. S. Litov, Ya. M. Poin and B. N. Markov. *Geodard. Inst. Prikladn. Khim., Sbornik State 1939-39*, 43-7(1939); *Khim. Referat. Zhne*, 1940, No. 2, 89.—The first com. production of Al_2O_3 from Tikhvin bauxite contg. 13-15% SiO_2 is described. Fuse fine-ground bauxite with water and limestone, lixiviate, purify the $Al(OH)_3$, obtained, evap., and return the soda soln. to the circuit. Of greater interest is the new method of producing Al_2O_3 from nepheline contg. up to 20% bases and 80% Al_2O_3 , the remainder being SiO_2 . Nepheline is a waste product from flotation of spath. First flotation nepheline with limestone in a rotary furnace, lixiviate with a soda soln., remove the limestone- SiO_2 slime (used to produce cement), free the Na and K aluminates from SiO_2 , treat with CO_2 and sep. $Al(OH)_3$ from the soln. contg. Na_2CO_3 and K_2CO_3 . Nepheline thus gives Al_2O_3 , Na_2CO_3 , K_2CO_3 and cement. Al alums are obtained by treating nepheline with 75% H_2SO_4 and an alkali metal salt. Treating with KCl produces K alums in a 90% yield based on Al_2O_3 . The slime has bleaching properties and can be used to refine petroleum products or to make water glass. Al_2O_3 can be made by heating clay at 600-800°, treating with HNO_3 , freeing the $Al(NO_3)_3$ from Fe by activated MnO_2 , partly satg. with NH_3 , evap., and treating with NH_4OH . The $Al(OH)_3$ residue is calcined, the NH_4NO_3 soln. evapd. and crystd. W. R. Henn

439-51A METALLURGICAL LITERATURE CLASSIFICATION

FROM DIVISION

DATE

PROCESS AND PROPERTIES INDEX

Purifying Ni electrolyte. Ya. M. Pavin and O. I. Andreeva. Russ. 57,817, Aug. 31, 1940. The Ni soln. is treated at 80° with Ni(OH)₂ in a quantity equiv. to the Fe present, the Fe(OH)₃ formed is removed, and soln. treated with a further quantity of Ni(OH)₂ to remove Co.

4

ASB-51A METALLURGICAL LITERATURE CLASSIFICATION

SEARCHED	INDEXED	SERIALIZED	FILED	SEARCHED	INDEXED	SERIALIZED	FILED

6

Fractional purification of Ni electrolytes for the removal of Fe and Co. Ya. M. Loshakov, I. Andreeva, A. A. Moeno and M. P. Schmanzar. *Tsvetnye Metal.* 10, No. 8, 29-35 (1941); *Chem. Zvest.* 1943, 1, 1602. With NaClO and NiSO₄ in the presence of NaOH the reactions $\text{Ni(OH)}_2 + \text{NaClO} + \text{H}_2\text{O} = \text{Ni(OH)}_3 + \text{NaCl}$ and $\text{NiSO}_4 + 2\text{NaOH} = \text{Ni(OH)}_2 + \text{Na}_2\text{SO}_4$ occur. Increasing the amt. of NaClO from 0.5 to 2.0 equiv. raised the NiO content, increased the active O in the black hydrate. Further increase in the amt. of NaClO added had no further effect on the activity of the hydrate obtained. A max. of 45% of the hypochlorite was used for the oxidation of the Ni, the remainder being decompd. by the catalytic action of the black hydrate. Between 0° and 20° the reaction proceeded quietly. As the temp. was increased to 60° the reaction was more violent, and it was necessary to add the NaClO and NaOH carefully. Increasing the temp. decreased the amt. of active O in the black hydrate. Increasing the amt. of alkali at const. hypochlorite concn. increased the amt. of O in the black hydrate; thus, at 1.15 equiv. NaOH the product was NiO_{0.25} and at 10.0 equiv. it was NiO_{0.15}. Varying the concn. of NaClO and that of NiSO₄ had no effect on the activity of the black hydrate. Fractional addn. of the NaClO (0.1-0.5 equiv. per mol.) gave a hydrate higher in O than that obtained when the NaClO was added all at once. Fractional addn. of the NaClO likewise increased its utilization coeff. When Na₂CO₃ was used instead of NaOH the active O content of the hydrate was considerably lower. Optimum conditions for obtaining the black hydrate are: hypo-

chlorite 1.5 equiv. (added on the NiO), NaOH 1.1 equiv., room temp. and fractional addn. of the NaClO. The black hydrate thus obtained contained 60.5% active O. Washing the black hydrate with water decreased the activity by an amt. that depended on the temp. of the water and the amt. of the hydrate; ice water caused the least loss of activity. The loss was greater on washing the hydrate that was not freshly prepd. The addn. of alkali (5%) to the wash water stabilized the black hydrate.

M. G. Moore

A13-51A METALLURGICAL LITERATURE CLASSIFICATION

eA

6

Interaction between sulfur dioxide and copper sulfate solutions. Ya. M. Pesin and M. L. Shabashova (Leningrad Technol. Inst.). *Zhur. Priklad. Khim.* (J. Applied

Chem.) 23, 278-84 (1950).—The solid ppt. formed by the action of 100% SO_2 gas, flowing at a rate of 1.5 l./hr., on a 10% soln. of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, at 60° and at 80° , was identified (by analysis, crystallography, and x-ray diffraction) as Chevrel's salt $\text{CuSO}_4 \cdot \text{Cu}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ (I). Consequently, the reaction equation at 60° and 80° is $3 \text{CuSO}_4 + 3 \text{H}_2\text{SO}_4 + 3 \text{H}_2\text{O} = \text{I} + 4 \text{H}_2\text{SO}_4$. At 100° , the solid is entirely metallic Cu, in conformity with the reaction $\text{CuSO}_4 + \text{H}_2\text{SO}_4 + \text{H}_2\text{O} = \text{Cu} + 2 \text{H}_2\text{SO}_4$. Decompn. of I (2 g. in 300-400 ml. H_2O) on boiling, under a stream of CO_2 , proceeded according to $3 \text{I} = 5 \text{SO}_2 + \text{CuSO}_4 + 4 \text{Cu}_2\text{O}$. N. Thom

b

CA

The interreaction between sulfur dioxide and copper sulfate solutions. Ya. M. Prain and M. L. Stakshova (Leningrad Inst. Technol.). *J. Appl. Chem. U.S.S.R.* 23, 289-95(1950) (Engl. translation).—See *C.A.* 43, 2900i.
R. M. S.

6

CA

The oxidation of $\text{CuSO}_4 \cdot 2\text{H}_2\text{O}$ to CuSO_4 . Ye. M. Pechu and M. I. Bhabashova (Leningrad Technol. Inst.). *Zhur. Priklad. Khim.* (L. Applied Chem.) 23, 350-6 (1950).—The formation of H_2SO_4 in the passage of a mixt. of SO_2 (7%) and air through a soln. of $\text{CuSO}_4 \cdot 2\text{H}_2\text{O}$ and 2.31% Cu at 60° and 80° is retarded as H_2SO_4 accumulates in the soln. owing to formation of CuSO_4 , Cu_2SO_4 , and Cu_3SO_4 . The soly. of that salt, in H_2O and in 10, 20, and 30% $\text{CuSO}_4 \cdot 2\text{H}_2\text{O}$, was detd., at 25°, to be 0.042, 0.058, 0.080, and 0.100 wt. %, and at 60°, to be 0.140, 0.312, 0.334, and 0.379 wt. %. The increase of the soly. with increasing CuSO_4 content of the soln. is distinct, but not sufficient to make practical the process of extn. of Cu by passing SO_2 gas into a CuSO_4 soln. The extn. can be effected by oxidation of I to CuSO_4 . In an aq. suspension of 4 g. I in 100 ml. H_2O , passage of air at 60° produced a solid phase of the empirical compn. $2\text{CuI} \cdot \text{CuSO}_4 \cdot 2\text{H}_2\text{O}$ (or $2\text{Cu} \cdot \text{OII} \cdot \text{CuSO}_4$) and a soln. with the mol. ratio $\text{Cu} : \text{S} = 1$. The process, consequently, is $2\text{I} + 3\text{O}_2 = 2\text{Cu}(\text{OII}) \cdot \text{CuSO}_4 + 2\text{H}_2\text{O}$. In a 10% H_2SO_4 soln., all of the I was converted to CuSO_4 . Complete oxidation of I and its soln. in the form of CuSO_4 was obtained by passing a mixt. of SO_2 with excess O_2 (air), the overall reaction being $\text{I} + \text{SO}_2 + 2\text{H}_2\text{O} = 3\text{CuSO}_4 + 2\text{H}_2\text{O}$. The rate of oxidation of I increases with the O_2 content in the gas and with the CuSO_4 content of the soln.; Cu^{2+} ions catalyze the oxidation $2\text{SO}_2 + \text{O}_2 + 2\text{H}_2\text{O} = 2\text{H}_2\text{SO}_4$.

009

18

Production of copper sulfate from copper oxide and gases containing sulfur dioxide and oxygen. V. M. Krain and M. L. Shastanova (Leningrad Technol. Inst.). *Zhur. Priklad. Khim.* (J. Applied Chem.) 33, 480-9 (1950).—The practical possibility of extn. of Cu, in the form of CuSO_4 in soln., from CuO , by contact with $\text{SO}_2 + \text{O}_2$, was demonstrated with 3 kinds of CuO , one prepd. by oxidation of Cu_2O at $500-600^\circ$ (79.8% Cu), the 2nd by oxidation of Cu powder with iron scrap (79.93% Cu), a 3rd by oxidation of Cu at $600-700^\circ$ (80.3% Cu). With a gas contg. 7% SO_2 , passed at the rate of 10 l./hr. at $85-93^\circ$, extn. of Cu was complete with all 3 samples; e.g., with the 3rd sample, after 0, 60, 120, and 180 min., the $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ content of the soln. was 0.58, 13.79, 16.41, and 17.98%, the degree of extn. of Cu from CuO , 0, 38.3, 81.3, and 100%, free H_2SO_4 , 0, 0.34, 0.22, and 0.10%. The solid phase, originally CuO , undergoes visible changes before it is dissolved. By analyses and x-ray examn., the solid phase is a mixt. of Cu_2O and $\text{CuSO}_4 \cdot \text{Cu}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ (I), the amt. of the latter increasing with time. As an example, after 0, 20, 40, and 60 min., the compn. of the solid phase (CuO , Cu_2O , I) was: 100, 0, 0; 0, 61.93, 38.12; 0, 32.5, 67.5; and 0, 2.29, 97.71%. The 1st product of the reaction between SO_2 and CuO is I which, in H_2O , reacts further along $3\text{I} + 18\text{CuO} \rightarrow 6\text{CuSO}_4 + 9\text{Cu}_2\text{O} + \text{water}$; the oxidation of I proceeds according to $\text{I} + \text{SO}_2 + 2\text{O}_2 = 3\text{CuSO}_4 + 2\text{H}_2\text{O}$. The overall process involves, simultaneously, direct soln. of CuO in the H_2SO_4 formed by oxidation of SO_2 to SO_3 in the presence of Cu^{++} ions, and formation of I which is further converted to CuSO_4 . The latter is produced about 2 to 3 times as fast as in the absence of Cu^{++} ions. The tech. process can be conducted with gases contg. as low as 1-4% SO_2 , provided the O_2 content is high enough to give a vol. ratio $\text{O}_2:\text{SO}_2$ not below 4. The coeff. of utilization of Cu is 94-75%.

16. Thon