"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHEMYAKIN, M.M.; SHCHUKINA, L.A.; VINOGRADOVA, Ye.I.; KOLOSOV, M.N.; VDOVINA, R.G.; KARAPETYAN, M.G.; RODIONOV, V.Ya.; RAVDEL', G.A.; SHVETSOV, Yu.B., BAMDAS, E.M.; CHAMAN, Ye.S.; YERMOLAYEV, K.M.; SEMKIN, Ye.P.

Research data on sarkomycin and its analogues. Part 1: Synthesis of dihydrosarkomycin and its antipode. Zhur. ob. khim. 27 no.3:742-748 Mr '57. (MLRA 10:6)

1. Institut biologicheskoy i meditsinskoy khimii Akademii meditsinskikh nauk SSSR.

(Sarkomycin)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SPIRICHEV, V.B., TSI CHZHEN-U [Ch'i Cheng-wu], OREKHOVICH, V.N., SHCHUKINA, L.A.

Reversible action of acylase. Report No.1: Enzymatic hydrolysis and synthesis of L-acetylalanine [with summary in English]. Biokhimiia 23 no.6:895-898 N-D '58 (MIRA 11:12)

1. Institut biologicheskoy i meditsinskoy khimii AMN SSSR, Moskva.
(ACYLASE)
(ALANINE)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

AUTHORS: Spirichev, V. B., Shchukina, L. A. SOV/79-28-6-62/63

TITLE: The Synthesis of l-Acetylalanine From d,l-Alanine (Sintez l-atsetilalanina iz d,l-alanina)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 6, pp. 1709-1709 (USSR)

ABSTRACT: As is known one of the most convenient preparative methods for the separation of racemic amino acids into the antipodes is that by Greenstein and collaborators (Refs 1,2). This method is based on the capability of acylase to deacylate only the l-forms of various N-acylated α-amino acids, while the d-forms of the acylamino acids remain practically untouched. The authors showed that the deacylation process of the amino acids by acylase can be inversed and that on certain conditions by its means a synthesis of l-acylamino acids from l- or d,l-amino acids can be made possible. Thus d,l-alanine by acylase obtained from pork-kidneys (Refs 2,3) in the course of 24 hours in the presence of 2 moles of sodium acetate converts to l-acetylalanine in a yield of 15%. The acylation capability of acylase which was found

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The Synthesis of l-Acetylalanine From d,l-Alanine SOV/79-28-6-62/63

by the authors can serve as basis for the method of synthesis of l-acylamino acid directly from racemic amino acids. There are 3 references, 1 of which is Soviet.

ASSOCIATION: Institut biologicheskoy i meditsinskoy khimii Akademii meditsinskikh nauk SSSR (Institute of Biological and Medical Chemistry, Academy of Medical Sciences USSR)

SUBMITTED: February 24, 1957

1. Alanines--Synthesis

Card 2/2

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

OREKHOVICH, V.N.; SHCHUKINA, L.A.; TSI CHZHEN-U [Ch'i Cheng-wu]; SPIRICHEV, V.B.

Role of the nature of amino acid and acyl radicals in enzymatic synthesis of l-acyl-amino acids by acylase I. Biokhimiia 24 no.4:667-671 Jl-Ag '59. (MIRA 12:11)

1. Institut biologicheskoy i meditsinskoy khimii Akademii meditsinskikh nauk SSSR, Moskva.
(ESTERASES chem.)
(AMINO ACIDS chem.)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

5(3)

AUTHORS: Shemyakin, M. M., Shigorin, D. N., Shchukina, L. A., Semkin, Ye. P.

SOV/62-59-4-20/42

TITLE: Structure and Mechanism of the Hydrolytic Splitting of α-Nitro-α-Phenylacetophenon o-Carboxylic Acid (Stroyeniye i mekhanizm gidroliticheskogo rasshchepleniya α-nitro-α-fenil-atsetofenon-o-karbonovoy kisloty)

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 4, pp 695-698 (USSR)

ABSTRACT: To determine the structure of α-nitro-α-phenylacetophenone-o-carboxylic acid and its salts the spectra of these compounds were investigated in the present work (Table 1). These investigations have provided an answer to the question relating to their structure and their different behavior in the presence of hydrolyzing agents. As was to be expected, α-nitro-α-phenylacetophenone-o-carboxylic acid, like other aromatic o-aldehyde-(keto)-acids, has the structure of lactol (IIIb) rather than that of the keto acid (IV) in the crystalline state as well as in solution. After the actual structure of the α-nitro-α-phenylacetophenonic acid and of its disodium salt had been clarified, its different behavior in the

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

Structure and Mechanism of the Hydrolytic Splitting of SOV/62-59-4-20/42
α-Nitro-α-Phenylacetophenon -o-Carboxylic Acid

presence of hydrolyzing agents has been understood. As was shown before (Ref 3) the C—C bonds can split in those compounds in which a prototropic group (V) is present or can be formed in the molecule. The tendency to split depends directly on the degree of polarization of the C—C bond under the action of the substituent. α-Nitro-dinitrophenylacetophenone-o-carboxylic acid itself, having a lactol (IIIb) structure, does not only contain the required group (V) but also a nitro group which can polarize the splitting bond to a very high degree in the required direction. For this very reason the acid (IIIb) splits easily to form phthalic acid anhydride and phenylnitromethane if the pH-value of the solution exceeds 7. In the molecule of the disodium salt, on the other hand, the prototropic group (V) is not contained nor can it be formed by hydration owing to the structure of this salt. This fact is responsible for the resistance of this compound to hydrolytic splitting. There are 1 table and 11 references, 8 of which are Soviet.

Card 2/3

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

Structure and Mechanism of the Hydrolytic Splitting of SOV/62-59-4-20/42
α-Nitro-αPhenylacetophenon -o-Carboxylic Acid

ASSOCIATION: Institut biologicheskoy i meditsinskoy khimii Akademii meditsinskikh nauk SSSR (Institute of Biological and Medical Chemistry of the Academy of Medical Sciences, USSR)

SUBMITTED: July 13, 1957

Card 3/3

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SOV/79-29-1-74/74

AUTHORS: Shchukina, L. A., Kara-Murza, S. N., Vdovina, R. G.

TITLE: Synthesis of O-Peptides With Help of N,N'-Dicyclohexyl Carbodiimide (Sintez O-peptidov s pomoshch'yu N,N'-ditsiklogeksilkarbodiimida)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 1, p 340 (USSR)

ABSTRACT: The synthesis of O-peptides of β-oxy-α-amino acids is of great interest as such compounds are biochemically of great importance. In many cases they are difficult to synthesize. The authors succeeded in bringing about a simple synthesis of O-peptides which owes its existence to N,N'-dicyclohexyl carbodiimide in the condensation of esters of the N-acylated oxyamino acids with N-acylamino acids. The reaction proceeds in the presence of pyridine in acetone (or in other organic solvents) at 20° in the course of 24 hours. Thus, the following products were obtained: 1) From the ethyl ester of N-benzoyl-seryl glycine and carbobenzoxyl leucyl the ethyl ester of O-carbobenzoyl leucyl-N-benzoyl-seryl glycine (yield: 84%). 2) From the ethyl ester of N-benzoyl-seryl glycine and carbobenzoxy-phenyl alanine of the ethyl ester of O-carbobenzoxy

Card 1/2

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SOV/79-29-1-74/74

Synthesis of O-Peptides With Help of N,N'-Dicyclohexyl Carbodiimide

phenyl alanyl-N-benzoyl-seryl glycine (yield: 82%). Apart from this under similar conditions from the amide of salicyclic acid and carbobenzoxy-phenyl alanine the amide of O-carbobenzoxy-phenyl analyl salicylic acid were obtained (yield 85%). There is 1 reference.

ASSOCIATION: Institut biologicheskoy i meditsinskoy khimii Akademii meditsinskikh nauk SSSR (Institute for Biological and Medical Chemistry of the Academy of Medical Sciences, USSR)

SUBMITTED: September 1, 1958

Card 2/2

USCOMM-DC-60,660

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, L. A., Doc Chem Sci -- (diss) "Oxidative and hydrolytic reactions of organic compounds." Moscow, Academy of Sciences USSR Publishing House, 1960. 24 pp; (Academy of Sciences USSR); 225 copies; free; list of author's work on pp 23-24 (21 entries); (KL, 24-60, 128)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, L.A.

Mechanism of 2-phenyl-2-hydroxy-1,3-indandione formation from dibromobenzalphthalide. Zhur.ob.khim. 31 no.9:3041-3045 S '61. (MIRA 14:9)

1. Institut biologicheskoy i meditsinskoy khimii Akademii meditsinskikh nauk SSSR.
(Indandione) (Phthalide)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, L.A.; KARA-MURZA, S.N.; GROMOVA, G.F.

New method of preparing S-aminoacyl derivatives of cysteine peptides. Dokl. AN SSSR 136 no.6:1351-1353 F '61. (MIRA 14:3)

1. Institut biologicheskoy i meditsinskoy khimii AMN SSSR. Predstavleno akademikom M. M. Shemyakinym.
(Cysteine)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

RAVDEL', G.A.; KRIT, N.A.; SHCHUKINA, L.A.; SHEMYAKIN, M.M., akademik

Synthetic paths in the preparation of the peptide part of ergot alkaloids. Dokl.AN SSSR 137 no.6.1377-1380 Ap '61. (MIRA 14:4)

1. Institut boilogicheskoy i meditsinskoy khimii Akademii meditsinskikh nauk SSSR.
(Ergot alkaloids)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHEMYAKIN, M.M., akademik; VINOGRADOVA, Ye.I.; FEYGINA, M.Yu.; ALDANOVA, N.A.; OLADKINA, V.A.; SHCHUKINA, L.A.

Synthesis of optically active depsipeptides. Dokl. AN SSSR 140 no.2:387-390 S '61. (MIRA 14:9)

1. Institut khimii prirodnykh soyedineniy AN SSSR.
(Peptides)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

ANTONOV, V. K., Institute for Chemistry of Natural Compounds, Academy of Sciences USSR, Moscow - "Transporic transformations of hydroxyacylcyclopeptides" (Section III)

BENEVITSKAYA, V. A., Institute for Chemistry of Natural Compounds, Academy of Sciences USSR, Moscow - "Synthesis and chemical behavior of model glycopeptides" (Section III)

SHCHUKINA, L. A., Institute for Chemistry of Natural Compounds, Academy of Sciences USSR, Moscow - "Synthesis of cyclic dipsipeptides" (Section III)

reports to be submitted for the Fifth European Peptide Symposium, Oxford, England, 3-7 Sep 1962.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, L.A.; VDOVINA, R.G.; SHVETSOV, Yu.B.; KARPOVA, A.V.

Preparative method of production of L- and D-α-hydroxyisovaleric acid. Izv. AN SSSR Otd.khim.nauk no.2:310-312 F '62. (MIRA 15:2)

1. Institut khimii prirodnykh soyedineniy AN SSSR i Institut biologicheskoy i meditsinskoy khimii AMN SSSR.
(Isovaleric acid)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, L.A.; SEMKIN, Ye.P.

Oxidative and oxidative-hydrolytic transformations of organic molecules. Part 34: Synthesis, properties, and hydrolytic transformations of halo- and hydroxytriketones of the tetralin series. Zhur.ob.khim. 32 no.2:473-483 F '62. (MIRA 15:2)

1. Institut biologicheskoy i meditsinskoy khimii AMN SSSR.
(Naphthalene)
(Ketones)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, L.A.; SEMKIN, Ye.P.

Oxidative and oxidative-hydrolytic transformations of organic molecules. Part 35: Synthesis and properties of polyfunctional substituted indans. Zhur.ob.khim. 32 no.2: 483-493 F '62. (MIRA 15:2)

1. Institut biologicheskoy i meditsinskoy khimii AMN SSSR. (Indan)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

ZHDANOV, G.L.; SHCHUKINA, L.A.; SOROKINA, I.B.; MAL'KOVA, V.P.; SEDOV, K.A.; RYABOVA, I.D.; SEMYIN, Ye.P.

Study of the biological activity of N-dichloroacetyl-D, L-serine. Dokl. AN SSSR 143 no.5:1222-1224 Ap '62. (MIRA 15:4)

1. Institut khimii prirodnykh soyedineniy AN SSSR. Predstavleno akademikom M.M.Shemyakinym.

(Serine)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SUCHUKINA, L. A.; RAVDEL', G. A.

"Depsipeptide analogs of biologically active peptides."

report submitted for 7th European Peptide Symp, Budapest, 3-8 Sep 64.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, L. A.; ZHUZE, A. L.; SEMKIN, Ye. P.; KRASNOVA, S. N.

Depsipeptide analogs of biologically active peptides. Report No. 1: Synthesis of depsipeptide analogs of ophthalmic acid and glutathione. Izv AN SSSR Ser Khim no. 4:685-692 Ap '64. (MIRA 17:5)

1. Institut khimii prirodnykh soyedineniy AN SSSR.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

RAVDEL', G.A.; KRIT, N.A.; OLADKINA, V.A.; SHCHUKINA, L.A.; SHEMYAKIN, M.M.

Depsipeptides. Report No.31: Synthesis of depsipeptides containing α-hydroxy-β-amino acid radicals. Izv. AN SSSR. Ser. khim. no.11:1987-1992 '65. (MIRA 18:11)

1. Institut khimii prirodnykh soyedineniy AN SSSR.

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, L. S.

SHCHUKINA, L. S.

"Investigation in the Group of Vitamin K. I. Synthesis of Potassium 2-methyl-1, 4-Napthoquinone-3-Sulphonate". Botchvar, D. A., Shchukina, L. S. Schernyshev, A. S., Semenova, N. G. and Shemiakin, M. M. (p. 326)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1943, Volume 13, no. 4-5.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

GRIGOR'YEVA, V.A. [Hryhor'ieva, V.A.]; RADZIYEVSKIY, A.R. [Radziievs'kyi, O.R.]; SHCHUKINA, L.V.

On biochemical muscular changes in insufficient blood supply. Ukr. biokhim. zhur. 36 no.2:258-266 '64. (MIRA 17:11)

1. Institute of Biochemistry of the Academy of Sciences of the Ukrainian S.S.R., Kiyev.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

FERDMAN, D.L.; GRIGOR'YEVA, V.A.; RADZIYEVSKIY, A.R.; SHCHUKINA, L.V.

Effect of adenosine triphosphate on the course of biochemical processes in the muscles in circulatory disorders. Klin. khir. no.2:29-33 '65. (MIRA 18:10)

1. Institut biokhimii AN UkrSSR (dir.- akademik A.V. Palladin) i Institut zoologii AN UkrSSR (dir.- doktor biolog. nauk P.M. Mezhuga).

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M.; BABENKOVA, K.; SHARONOV, V.

Let's align with the best. Okhr. truda i sots. strakh. 5 no.8:20-21 Ag '62. (MIRA 15:7)

1. Strakhovyye delegaty chasovogo zavoda, g. Orel.
(Orel--Clockmaking and watchmaking--Hygienic aspects)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. A.

SHCHUKINA, M. A. -- "The Methods of Applying and Systematizing Geometric Knowledge of the 'Polyhedron' Subject in the Tenth Class." Min Education RSFSR, Leningrad State Pedagogical Institute imeni A. I. Hersen, Chair of Elementary Mathematics, Leningrad, 1956. (Dissertation for the Degree of Candidate of Pedagogical Sciences)

SO: Knizhnaya Letopis' No 43, October 1956, Moscow

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. A.

Use of previously adopted geometrical knowledge by students of the 10th grade (on the subject "polyhedra"). Uch. zap. Ped. inst. Gerts. 183:335-355 '58. (MIRA 13:8)

(Polyhedra)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

ABUGOVA, Khaya Beniaminova; SHCHUKINA, Mariya Alekseyevna; PAZEL'SKIY, S.V., red.; KOZLOVSKAYA, M.D., tekhn.red.

[Collection of oral exercises in geometry for high school, grades 8-10] Sbornik ustnykh uprazhnenii po geometrii dlia 8-10 klassov srednei shkoly; posobie dlia uchitelei srednei shkoly. Moskva, Gos.uchebno-pedagog.izd-vo M-va prosv. RSFSR, 1960. 111 p. (MIRA 14:4)

(Mathematics--Problems, exercises, etc.)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M.I.

Pharmaceutical congress in Karlsbad. Med.prom. 14 no.1:57-58 Ja '60. (MIRA 13:5)

(PHARMACY--CONGRESSES)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M.N., prof.

Antitubercular medicinal preparations. Zhur. VKHO 10 no. 6: 637-648 '65 (MIRA 19:1)

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

BC

H-3

[redacted]

COMMON VARIABLES INDEX

COMMON ELEMENTS

MATERIALS INDEX

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

MATERIALS INDEX

3RD AND 4TH ORDERS

2ND LETTER

AUTHOR INDEX

1ST AND 2ND LETTER

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

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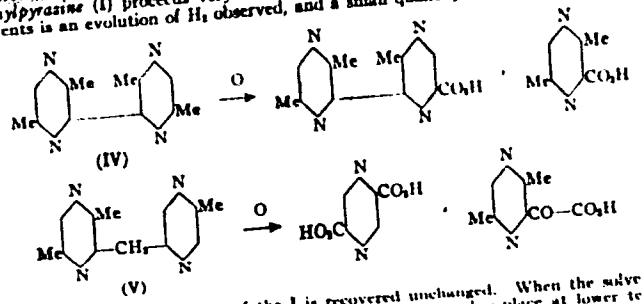
ca

The action of sodium amide on 2,5-dimethylpyrazine. A. K. Chichibabin and M. N. Shchukina, *J. Russ. Phys.-Chem. Soc.* 62, 1189-99(1930). Ammonia of 2,5-dimethylpyrazine (I) proceeds very slowly with $NaNH_2$. Only with strong heating in solvents is an evolution of H_2 observed, and a small quantity of 3-amino-2,5-dimethyl-



(IV)

(V)

pyrazine (II) isolated. Most of the I is recovered unchanged. When the solvent is omitted, higher mol. products are obtained, the reaction takes place at lower temps. and no H_2 is evolved. Under these conditions were isolated from the reaction mixt. some unchanged I, a small quantity of *dimethylpiperazine* (III), and 2 cryst. bases of the same empirical compn. (IV), m. 68°, and (V), m. 135°. The structures were detd. by oxidation with $KMnO_4$. In the formation of the homologous bipyrazyls, polymerization to tetramethylbipyrazyl occurs first, as is shown by the blue color of the salts formed by adding acids. As in the case of the analogous biquinolyls the color is destroyed by atm. O_2. The formation of V has no analogy in the pyridine and quinoline series. It probably arises according to the following mechanism, involving the tautomeric form of I:

OVER

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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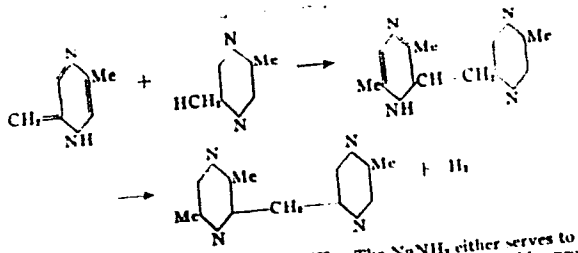

Some of the H_2 evidently goes to reduce I to III. The $NaNH_2$ either serves to produce the tautomer of I or else brings about the formation of intermediary addn. products to the pyrazine nucleus. In solvents, *e. g.*, xylene, above 145°, II was obtained in 10% yield and I was recovered to the extent of 80%. II, recrystd. from PhH, formed colorless needles, m. 111°, b_{10} 119°, sol. in H_2O, EtOH, Et_2O, $CHCl_3$, PhH and PhMe. Bz deriv., m. 100°. In 1 expt. without solvent, 20 g. I and 14.5 g. $NaNH_2$ gave after Et_2O extn. of the reaction products (4 hrs. on the H_2O bath) a red oil. This was fractionated, giving a low-boiling fraction consisting of I and II, and 8 g. of a mixt. of IV and V, b_{10} 160–75°. Further fractionation and crystn. from EtOH gave IV, sol. in EtOH, and V, sol. only in boiling EtOH. IV was recrystd. from petr. ether as colorless needles, m. 67.5–68.5°, b_{10} 107°, easily sol. in H_2O, EtOH, Et_2O, PhH, and $CHCl_3$, difficultly sol. in cold petr. ether. The aq. soln. is alk. to litmus. The soln. in strong HCl or H_2SO_4 is bright red. The $HgCl_2$ compd. was obtained as yellow prisms, m. 184° (decompn.). V crystd. from EtOH in long, colorless needles, m. 135°, b_{10} 158–7°. It dissolves less easily in cold H_2O, EtOH, Et_2O, PhH and ligroin than IV, but is easily sol. in $CHCl_3$. V gives no color with acids, and is neutral to litmus. The $HgCl_2$ compd. m. 224° (decompn.), and the $AgNO_3$ compd. m. 253° (decompn.).

Lewis W. Butz

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

1C

Synthesis of cocaine from hyoscyamine. M. N. Shchukina, R. A. Lapina and N. A. Preobrazhenskii. *Bull. acad. sci. U. R. S. S., Classe sci. math. nat., Sér. chim.* **1936**, 997-1004 (in German 1004). A new procedure for the prepn. of tropinonecarboxylic esters by the action of carboxylic esters on tropinone is given which runs smoothly at the m. p. of metallic Na or K with good yields and which permits the synthesis of cocaine from hyoscyamine and other tropines through tropine and tropinone. Tropine is obtained by simply heating hyoscyamine with H_2O. C. R. Addinall

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

a

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Separation of racemic scopolamine into the optical antipodes. M. N. Shchukina, S. S. Okun, D. N. Yurygin and N. A. Preobrazhenskii. *J. Gen. Chem.* (U. S. S. R.) 10, 803 8(1940).—The procedure for the sepn. of *l*-scopolamine (I) from the racemic mixt. formed in the process of com. extn. and reworking of I from the plant is based on the pptn. with *d*-camphoric acid, decompn. of the bi-*d*-camphorate of I with $NaHCO_3$ and extn. with $CHCl_3$. The mother liquor, contg. chiefly the *d*-isomer, is decompd. with $NaHCO_3$, extd. with $CHCl_3$, the residue from the solvent is treated in MeOH with MeONa and the racemate is treated as above. The operation can be repeated until nearly all of the I is sepd. Chas. Blanc

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CA

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4(5)-(Hydantoylmethyl)imidazole and its hydrolysis. M. N. Shchukina. *J. Gen. Chem.* (U. S. S. R.) 10, 1108-12(1940).—Attempts to repeat the work of Wada (*C. A.* 27, 3444) in getting histamine (I) from histidine (II) show that the reactions which he describes do not occur. II and urea condense when heated for 5 hrs. in H_2O to give a uramino acid which is converted during evapn. of the soln., acidified with HCl, to *4(5)-(hydantoylmethyl)imidazole-HCl* (III), m. 255°; *picrate*, m. 209°. The free base is an oil. Hydrolysis of III by $Ba(OH)_2$ for 1-2 hrs. gives *l*-II, and for 5 hrs. *dl*-II. III gives no reaction when heated with H_2SO_4. Contrary to the claims of W., I is not obtained in any of these reactions. H. M. Leicester

I. b. Alkaloids, Inst Org Chem, AS USSR + State Alkaloid Plant

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The products of condensation of α-formyl carboxylic esters with the esters of α-halo acids. II. Carboxylation of vinylalkyl esters. M. N. Shchukina and N. A. Preobrazhenskii. *J. Gen. Chem.* (U. S. S. R.) 10, 1363-8 (1940); cf. *C. A.* 30, 1030[8]. — $EtO_2CCHMeCHO$ (14 g.) was added to 2.5 g. Na in 50 cc. abs. alc. and the resulting mixt. was treated with 15 g. of CH_2ClCO_2Et. After heating in a water bath for 5 hrs. and distg. off the alc., the NaCl was dissolved in water and the $EtO_2CCMe:CHOCH_2CO_2Et$ (I) (11 g.) formed was extd. with ether and recovered in the usual way. I b_{12} 147-9°, b_{15} 153°, d_4^{20} 1.0038, n_D^{20} 1.4624, MR_D 54.36, sp. exaltation 0.692. Freshly distd. I decolorized Br water but did not color fuchsin-SO_2 and produced a mirror with Tollen's reagent only on boiling. Boiling I with water and $(CO_2H)_2$ yielded EtCHO which formed an aldol during the synthesis of the semicarbazone, yielding the aldol semicarbazone. The aq. soln. after sapon. of I contained $HOCH_2CO_2H$, identified as its hydrazide, m. 92°. The sapon. of I with NaOEt and alc.-water yielded $NaO_2CCH(CHO)Me$, identified as its Ba salt. The condensation (in similar manner) of $EtO_2CCHEtCH(CO_2Et)CHO$ (42.5 g.) with $EtCHBrCO_2Et$ (45 g.) yielded $EtO_2CCHEtC(CO_2Et):CHOCHEtCO_2Et$ (II) (42.5 g.), b_{12} 200-2°, oily liquid, decolorized Br water, produced a mirror with Tollen's reagent upon prolonged boiling. II hydrolyzed by boiling with dil. HCl yielded the hemialdehyde of ethylsuccinic acid and α-hydroxybutyric acid, m. 46°. The sapon. of II in NaOEt and alc.-water yielded a tribasic acid which split off CO_2 upon acidifying and formed $HO_2CCHEtCH:CHOCHEtCO_2H$, m. 148-50°. The dibasic acid distd. *in vacuo* partially formed $O.CO.CHEt.CH_2.CHOCHEtCO_2H$, b. 204-7°. The sapon. of $EtO_2CCH_2C(CO_2Et):CHOCHEtCO_2Et$ with NaOEt in alc.-water yielded $HO_2CCH_2CH:CHOCHEtCO_2H$, b_{14} 195-7°. A vacuum distn. of this acid yielded $O.CO.CH_2.CH_2.CHOCHEtCO_2H$. Treatment of $EtO_2CCHEtC(CO_2Et):CHOCH_2CO_2Et$ with $(CO_2H)_2$ yielded an acid, $C_8H_{12}O_5$, m. 102-3°, the titration of which disclosed that the acid had the formula $C_{10}H_{22}O_{10}$, $HO_2CCHEtCH:CHOCH_2CO_2CH(OCH_2CO_2H)CH_2CHEtCO_2H$. A. A. Podgorny

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Ca

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The action of acid chlorides on diethylhydroxylamine. A. Ya. Berlin, M. N. Shchukina, and E. D. Sazonova. *J. Gen. Chem.* (U.S.S.R.) 14, 249-56(1944)(English summary).—When Et_2NOH (I), KOH and $BzCl$ are stirred and cooled for 2 hrs. they give the normal product Et_2NOBz, an oil, purified as the H_2SO_4 salt, m. 133-4°. Hydrolysis with HCl regenerates the starting compds. With stronger acids, members of a new class, the amide oxides, are formed. I, $PhSO_2Cl$ and KOH must be stirred well and ice-cooled during the reaction, since if local heating occurs, violent decompn. and even explosion may result. When the reaction goes to completion, the product is $Et_2N(:O)SO_2Ph$ (II), which explodes when warmed and decomps. in H_2O to give AcH, $EtNH_2$, and $PhSO_2H$. In EtOH it gives $MeCH(OEt)_2$. It can be crystd. from Me_2CO and then decomps. 52°. Satn. of an Me_2CO soln. with SO_2 in the presence of a small amt. of H_2O gives *$MeCH(NHEt)OSO_2H$*, m. 124°, which easily hydrolyzes to AcH, $EtNH_2$, and H_2SO_4. I, CCl_3CO_2H and KOH forms the very unstable $Et_2N(:O)OCOCCl_3$, a non-crystg oil whose reactions are analogous to those of II.

H. M. Leicester

All-Union Sci-Res. Chemico-Pharmaceutical Inst. im Ordzhonikidze

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Ca

PROCESSES AND PROPERTIES INDEX

Preparation and properties of 2-bromovinyl alkyl ethers. A. E. Favorskii and M. N. Shchukina (Acad. Sci. U.S.S.R., Moscow). *J. Gen. Chem.* (U.S.S.R.) **15**, 385-93 (1945)(English summary); cf. *C.A.* **38**, 330[9].—Vinyl Et ether (36 g.) in 200 cc. $CHCl_3$ was treated with 72 g. Br at from −10° to −15° to yield 95 g. *1,2-dibromoethoxyethane* (I), b_{20} 80°, b_{10} 74°. I (35 g.) in 20 cc. MeOH was added slowly to 15.6 g. KOH in 50 cc. MeOH, heated to reflux for 5 min., and poured into ice water to give 70% *bromoacetaldehyde di-Me acetal*, b. 148-50°. Treatment with powd. KOH in Et_2O suspension led to complex products which appear to contain bromoacetal. I (75 g.), 50 g. Et_3NPh, and 150 cc. vaseline oil were heated on a steam bath for 1 hr. to yield 62% *BrCH:CHOEt*, b_{17} 35-45°, b. 137-40°, d_4^{20} 1.4137, n_D^{20} 1.4718. The ether is subject to partial decompn. on standing, yielding small amts. of EtOH and bromoacetal; it is stable to water and dil. alkalies, but is rapidly hydrolyzed by acids. It is readily polymerized to a clear glass on addn. of a crystal of $SnCl_4$, while addn. of $FeCl_3$ causes instantaneous polymerization to a brown crumbly mass. Distn. of the ether from fused KOH gave *ethynyl Et ether*, b. 49°, d_4^{20} 0.8001, n_D^{20} 1.3796. Bromination, as above, of 25.8 g. vinyl Pr ether gave 54 g. *1-(1,2-dibromoethoxy)propane*, b_{10} 86-7°, d_4^{20} 1.6130, n_D^{20} 1.4975; 17 g. of this, 10 g. Et_3NPh, and 80 cc. vaseline oil were heated on a steam bath for 2-3 hrs. to yield 5 g. *BrCH:CHOPr*, b_{10} 55-6°, b. 153-4° (slight decompn.), n_D^{20} 1.4630, d_4^{20} 1.3180. Vinyl Bu ether (20 g.) was treated at 10-15° with Br in 100 cc. $CHCl_3$, with only 16 g. Br being used before decolorization ceased; the reaction gave various by-products and about 18 g. *1-(1,2-dibromoethoxy)butane*, b_{10} 103-4°, n_D^{20} 1.4967, d_4^{20} 1.5524; heating 45 g. of this, 27.5 g. Et_3NPh, and 90 g. vaseline oil for 3 hrs. on a steam bath gave 13 g. *BrCH:CHOBu*, b_{10} 74°, n_D^{20} 1.4657, d_4^{20} 1.270. Iso-Pr vinyl ether (21 g.) in 100 cc. $CHCl_3$ was brominated with 40 g. Br with very good cooling which, however, could not prevent a temp. rise to 30° in the initial stage; the reaction was completed at 10° in the course of 3 hrs., since the Br addn. was very slow. Distn. gave aq. HBr, various org. by-products, and 12 g. *$Br_2CHCHBrOCHMe_2$*, b_{10} 118°, b_{18} 122-3°, d_4^{20} 1.9387, n_D^{20} 1.5332; only a small amt. of *1,2-dibromethyl iso-Pr ether*, b_{10} 77-9°, d_4^{20} 1.6424, n_D^{20} 1.4992, was obtained. Repetition of the expt. with 86 g. of the vinyl ether gave 32 g. of the di-Br ether. Dehydrohalogenation, as above, gave *2-bromovinyl isopropyl ether*, b_{10} 52°. G. M. K.

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1ST AND 2ND ORDERS — 3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

C2a

10

Preparation and properties of alkoxyacetylenes. A. E. Favorskii and M. N. Shchukina (Acad. Sci. U.S.S.R., Moscow). *J. Gen. Chem.* (U.S.S.R.) 15, 394–400(1945) (English summary).—A no. of alkoxyacetylenes were prepd. and were shown to be transformed into acetates under the influence of acids. It was shown that these acetylenic ethers add HCl to yield 1-chlorovinyl alkyl ethers, which readily undergo addn. reactions, but which have a rather firmly bound Cl atom which resists substitution. BrCH:CHOEt (20 g.) was placed in a flask equipped with a dephlegmator, the upper portion of which was filled with powd. K_2CO_3; 8 g. powd. KOH was added rapidly to the halo ether and the mixt. shaken and, after the vigorous reaction ceased, the product was slowly distd. There was obtained 70% *ethoxyacetylene* (I), b. 50°, n_D^{20} 1.3700, d_4^{20} 1.8001; *Ag salt*, white amorphous solid, explosive; *Cu salt*, bright yellow. Shaking the ether with acidified water (HCl, AcOH or H_2SO_4) leads to a vigorous reaction, with disappearance of the characteristic odor of the material and appearance of EtOAc odor; use of alc. HCl leads to formation of EtOAc and EtCl. I (8.4 g.) in 20 cc. dry Et_2O was treated with the theoretical amt. of HCl in dry Et_2O with cooling to yield 5.4 g. *1-chlorovinyl Et ether*, b. 88–90°, n_D^{20} 1.4208, d_4^{20} 1.0037; this reacts explosively with water to yield EtOAc; it is unreactive even on refluxing with dry EtONa. In a similar manner, 78% *propoxyacetylene* was obtained from BrCH:CHOPr; it b. 75°, n_D^{20} 1.3935, d_4^{20} 0.8980; *Ag salt*, darkens very rapidly; *Cu salt*, light-yellow powder. Treatment of the ether with dil. HCl leads to rapid formation of PrOAc; use of PrOH-HCl, however, followed by refluxing, led to a mixt. that retained the strong odor of the acetylene ether, which was made to disappear only after addn. of concd. H_2SO_4 (this was true in case of sufficient amts. of HCl-PrOH; if the latter were present in sufficient amts., substantially quant. formation of PrCl and PrOAc was observed). Treatment of the ether with Et₂O-HCl gave readily the *1-chlorovinyl Pr ether*, b. 112–14°, d_4^{20} 0.9825, n_D^{20} 1.4230. Similarly to the above, it b. 102–4°, n_D^{20} 1.4010, d_4^{20} 0.8184; it readily forms BuOH on shaking with acidified water; its *Cu salt* is a bright yellow powder.

G. M. Kosolapoff

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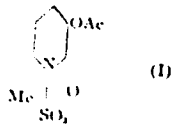
Cd

1ST AND 2ND ORDERS — PROCESSES — PROPERTIES INDEX — 3RD AND 4TH ORDERS

10

Arecoline N-oxide (genarecoline). M. N. Shchukina, A. Ya. Berlin, and E. D. Sazonova (All Union Chem. Pharm. Research Inst., Moscow). *J. Applied Chem. (U.S.S.R.)* 18, 631-7(1945). Arecoline HBr (5 g.) was converted into the free base by treatment with satd. K_2SO_4 with cooling. The Et_2O ext. of the mixt., after drying, was added slowly with cooling to 50 cc. Et_2O soln. of Bz_2O_2 contg. 0.025 atom of active O at 0-3°. The mixt. was then treated with 4.6 g. picric acid and allowed to stand for 2 hrs. to yield 6 g. *arecoline N-oxide picrate*, m. 118° (crude), 123° (from EtOH). The picrate (10 g.) was stirred with cooling with 80 cc. concd. HCl for 1 hr. after which the picric acid was filtered off, the filtrate extd. with Et_2O, and the aq. soln. evapd. *in vacuo* at 30°. The residue was dried at 50° *in vacuo*, and after extn. with several portions of $CHCl_3$ was vacuum-dried at 50° to yield 4 g. *arecoline N-oxide-HCl*, m. 141° (from abs. EtOH). Treatment of this with 25% K_2CO_3 with cooling gave the *free base* as a yellowish oil (from the $CHCl_3$ ext.), which is sol. in $CHCl_3$, difficultly sol. in Et_2O. Treatment of the HCl salt with SO_2 in water with ice-cooling gave *arecoline sulfamate* as shiny needles (it is difficultly sol. in cold water and alc., and hydrolyzes on warming to yield arecoline sulfate), m. 162° (from MeCOEt). The mother liquor from this prepn. contained arecoline which was isolated as the oxalate. The indications are that SO_2 effects the reduction of the oxide to the free base, the sulfamic ester (I) being an intermediate.

G. M. Kosolapoff



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Dichlorodiphenyltrichloroethane. M. N. Shchukina, A. Ya. Berlin, and E. D. Sazonova. U.S.S.R. 67,326, May 31, 1946. The reaction involves the condensation of PhCl with CCl_3CHO in the presence of fuming H_2SO_4. For the purposes of this reaction the CCl_3CHO is not sepd. from the reaction mixt. obtained in the chlorination of EtOH. When the reaction mixt. attains a sp. gr. of 1.56 at 30°, it is used directly for condensation with PhCl in the presence of fuming or concd. H_2SO_4, and the condensation product is sepd. in the usual manner.

M. Hosch

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SHCHUKINA, M. N. Dr. Chem. Sci.

Dissertation: "Investigations in the Field of Aliphatic Alpha-Chlorine and Alpha-Bromine Aldehydes." Inst of Organic Chemistry, Acad Sci USSR, 13 Feb 47.

SO: Vechernyaya Moskva, Feb, 1947 (Project #17836)

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1ST AND 2ND ORDERS — 3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

CA

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Structure and properties of α,β-dibromoalkyl ethers, β-bromovinyl ethers, and alkoxyacetylenes. M. N. Shchukina. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 18, 1350-60(1948). The properties of α,β-dibromoalkyl ethers are best explained by superposition of covalent and heteropolar states: RCHBrCHBrOEt ⟷ (RCHBr·CHOEt ⟷ RCHBrCH:OEt)⁺Br⁻. The reactivity of β-halo ethers and vinyl alkyl ethers is conditioned by the resonance in the ethoxycarbonium structure. The reactivity of alkoxyacetylenes and alkoxyethylenes is explained by their activation by proton addn. to the neg. end of the polar chain. The α,β-dibromoalkyl Et ethers were prepd. by Shoemaker and Boord technique (C.A. 25, 2681). Iso-BuCHO (26 g.) and 14 g. EtOH satd., with cooling, with 15 g. HCl and the product (33 g.) treated with 364 g. Br at 0°, gave 59 g. *1,2-dibromoisoamyl Et ether*, b_{15} 92-3°, d_4^{20} 1.5178, n_D^{20} 1.5012; t.as (14 g.) and 10 ml. MeOH treated with 6 g. KOH in 20 ml. MeOH and refluxed 0.5 hr. gave 70% *α-bromoisovaleraldehyde Me Et acetal*, b_{12} 76°, d_4^{20} 1.2250, n_D^{20} 1.4540. The procedure gave 70-80% yields of other acetals which are described in the literature: di-Me or di-Et acetals of $BrCH_2CHO$, $MeCHBrCH(OEt)_2$, and $EtCHBrCH(OEt)_2$. MeCHBrCHBrOEt (79 g.), 53 g. Et_3NPh, and 120 g. vaseline were heated 3 hrs. to 100°, cooled, dild. by Et_2O, and filtered, giving 57% *MeCBr:CHOEt*, b_{6-9} 45-7°, d_4^{20} 1.319, n_D^{20} 1.4683; it decolorizes Br water, reduces NH_3-$AgNO_3$ on heating, and gives a ppt. on shaking with 2% HgCl with liberation of Cl ions; addn. of alkali results in rapid deposition of metallic Hg. Treatment with a dil. HCl soln. of 2,4-$(O_2N)_2C_6H_3NHNH_2$ gives *α-ethoxypropionaldehyde 2,4-dinitrophenylhydrazone*, m. 126° (from EtOH). Heating 100 g. EtOCHBrCHBrCH₃Me, 150 g. vaseline, and 100 g. Et_3NPh gave in 3 hrs. at 102-5° 57% *1-ethoxy-2-bromo-1-butene*, b_{10} 64°, d_4^{20} 1.270, n_D^{20} 1.4926, which with $(O_2N)_2C_6H_3NHNH_2$ gives *α-ethoxybutyraldehyde 2,4-dinitrophenylhydrazone*, m. 82°. Similarly, EtOCHBrCHBrCHMe₂ gives *MeCHCBr:CHOEt*, b_{18} 67-70°, d_4^{20} 1.2025, n_D^{20} 1.4761, as a by-product, some *α-bromoisovaleraldehyde*, b_{10} 25-35°, was also obtained; the former compd. gives the *α-ethoxyisovaleraldehyde 2,4-dinitrophenylhydrazone*, m. 130-1° (from EtOH). EtOCH:CBrMe (32 g.) and 60 g. powd. KOH heated to 120° gave 61% *1-ethoxy-2-methylacetylene*, b. 84-5°, d_4^{20} 0.8276, n_D^{20} 1.4130, which on shaking with dil. acids evolves much heat and forms $EtCO_2Et$. EtOCH:CBrEt (30 g.) heated with 60 g. powd. KOH at 100-20° gave *1-ethoxy-2-ethylacetylene* (6 g.) in 2 cycles, b. 110-12°, d_4^{20} 0.8631, n_D^{20} 1.4196, which with dry HCl in EtOH yields EtCl and $PrCO_2Et$. Heating 16 g. EtOCH:CBrCHMe₂ with 32 g. KOH gave 3.8 g. *1-ethoxy-2-isopropylacetylene*, b. 125-5°, b_{10-5} 75-80°, d_4^{20} 0.8432, n_D^{20} 1.4258, which on shaking with acidified water gives iso-$BuCO_2Et$. G. M. Kosolapoff

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Pk 30/49T9

USSR/Chemistry - Synthesis Sep 48
Chemistry - Hydrolytic Reaction

"Synthetic Reactions by Means of Ethoxyacetylenemagnesium Bromide," M. N. Shchukina, I. A. Rubtsov, All-Union Chem Phar Sci Res Inst imeni S. Ordzhonikidze, Moscow Inst Fine Chem Tech imeni M. V. Lomonosov, 8 pp

"Zhur Obshch Khimii" Vol XVIII, No 9 - p 1645-52

Condensation of ethoxyethynylmagnesium bromide with acetone, acetophenone and 3-buten-2-OH gave the corresponding tertiary alkoxyethynylcarbinols. The latter yield esters of β-oxyacids or α, β-unsaturated acids when treated with dilute acids. Hydrogenation first gives β-ethoxyvinylcarbinols (I) and later

[redacted] 30/49T9

USSR/Chemistry - Synthesis (Contd) Sep 48

saturated β-ethoxyalcohols. Hydrolysis of (I) gives β-oxyaldehydes or α, β-unsaturated aldehydes. Submitted 19 May 47.

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Chlorination and bromination of acetaldehyde and lower homologs M. N. Shchukina. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 18, 1653-62 (1948). Chlorination of AcH at 16-18° proceeds through a substance composed of 2 moles $ClCH_2CHO$, 1 mole AcH, and 1 mole HCl, which on distn. dissoc. and gives 60% $ClCH_2CHO$. Prolonged chlorination of AcH at 70-80° gives mainly Cl_3CHCHO, while at 80-90° chloral forms mainly, with some $MeCHClCCl_2CHO$. Bromination of AcH and homologs proceeds at a lower temp. than the chlorination and gives 1,2-di-Br ales. as primary products, further action is slow and requires a higher temp. The mechanism of chlorination and bromination must be regarded as consisting of proton addn. to give an intermediate which has a hydroxycarbenium structure in one of the equil. states, the presence of the incomplete electron octet explains the ease of halogenation and the polymerization tendency; the difficulty of continued reaction is explained by decreased wt. of the carbenium structure after introduction of 1 Br atom. Passage of dry Cl into 44 g. AcH leads to a temp. rise despite cooling and is best done at 16-18°, as an initially preset -5° temp. leads to violent action delayed 2-10 min.; HCl evolution starts after 24 g. Cl is taken up. When a 3 mols. AcH:2 mols. Cl ratio is reached, Cl is no longer absorbed; 13-14 g. HCl is recovered and the product, 78-84 g., is a fuming colorless liquid, whose analysis confirms the above-given complex. Distn. gives 13 g. HCl, 8 g. AcH, 47 g. *$ClCH_2CHO$*, b. 80-6° (*2,4-dinitrophenylhydrazone*, m. 158-9°), and 10 g. residue of *trichloroparaldehyde*, m. 83-4°. On standing 24 hrs. the complex seps. into 2 layers: upper, aq. HCl; and bottom, *$ClCH_2CHO.H_2O$*, b. 88-93°, b_{10} 23-5°. Addn. of 44 g. of the complex to 50 g. thiourea in water, followed by 28 g. $NaHCO_3$ and heating 2 hrs. at 85-90°, gave 48.50°, 2-aminothiazole, m. 96° (from benzene). If the chlorination is continued 12 hrs. with gradual heating to 70-80°, there is obtained 80 g. product, which on distn. gives 57.5°, *Cl_3CHCHO*, b. 88-90°, yielding, with $2,4-(O_2N)_2C_6H_3NHNH_2$ the corresponding glyoxal deriv., m. 315°; on standing, *Cl_3HCCHO* gives a solid *polymer* which dissociates on distn. Similar chlorination carried further 8 hrs. at 80°, 12 hrs. at 80-90°, and 8 hrs. at 90° (Cl absorption stops) gave 120 g. crude product yielding on distn. 50% *chloral*, followed by 9.5 g. *trichlorobutyraldehyde*, m. 78° (b. about 150°), addn. of Fe chloride (from 3 g. Fe and HCl) gives a somewhat lower yield of chloral. Bromination was conducted as described earlier (Stepanov, *et al.*, *C.A.* 21, 731) and is facilitated by illumination; the induction period is 2-3 min. and the addn. of Br must be halted until the initial action subsides. When 1 mol. Br is added at 5°, the addn. stops and addn. of ales. to this primary product gives α-bromoacetals; in this manner the di-Et acetals of $BrCH_2CHO$, $MeCHBrCHO$, $EtCHBrCHO$, and $Me_2CHCHBrCHO$ were obtained in 69-73% yields (no data). Addn. of a 2nd Br requires 6-7 hrs. at 25-40°; passage of dry N or CO_2 is used to remove the HBr prior to isolation of the products; treatment with P_2O_5 and distn. gave 55% *Br_2CHCHO*, b. 137-40°, 48% *$MeCBr_2CHO$*, b_{10} 128-30° (with semicarbazide-HCl this gives methylglyoxal disemicarbazone, m. 252-3°, and with $HC(OEt)_3$ 79% *$MeCBr_2CH(OEt)_2$*, b_{14} 96-8°, d_4^{18} 1.5513, n_D^{18} 1.4783), and 61% *$EtCBr_2CHO$*, b_{10} 85° (*di-Et acetal*, d_4^{18} 1.645, n_D^{18} 1.4875), from the corresponding aldehydes.

G. M. Kosolapoff

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. N.

PA 65/49T22

USSR/Chemistry - Chloral
Anilides

Apr 49

"The Condensation of Acylanilides With Chloral," A. Ya. Berlin, M. N. Shchukina, Ye. D. Sazonova, All-Union Sci Res Chemicophar Inst imeni S. Ordzhonikidze, 6 pp

"Zhur Obshch Khim" Vol XIX, No 4 - p. 639-45

Study of subject reaction in the presence of H_2SO_4 established that acetanilide and phthalanil enter into the reaction, while succinanil, in the observed experiments, did not. Gives products of the reaction of acetanilide and the phthalanil with the chloral.

[redacted]

65/49T22

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

CA

10

Hydroxy- and methoxyamino-substituted 2,2-diphenyl-1,1,1-trichloroethane. M. N. Shchukina and E. D. Sizonova (All-Union Research Inst. Pharm Chem., Moscow). *J. Gen. Chem. U.S.S.R.* **19**, No. 11, a565-70 (1949)(Engl. translation).—See *C.A.* **44**, 3952c.

E. J. C.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

Hydroxy and methoxyamino substituted derivatives of 2,2-diphenyl-1,1,1-trichloroethane M. N. Shchukina and E. D. Sazonova (All Union Chem. Pharm. Research Inst., Moscow). *Zhur. Obshchei Khim.* (J. Gen. Chem.), 19, 2004-9(1949). *p*-AcNHC$_6$H$_4$OMe (16.6 g.) stirred at 5° with 75 ml. 100% H_2SO_4 and 7.5 g. Cl_3CCHO, then 0.5 hr. at room temp., gave 13 g. [2,5-*MeO*(*AcNH*)-C_6H_3]$_2$*CHCCl*$_3$ (I), m. 226.7° (from EtOH), contg. EtOH of crystn.; boiling with dil. HCl 6 hrs. gave the free *amino deriv.*, m. 74°; *di-HCl salt*, m. above 230° (from EtOH). *o*-AcNHC$_6$H$_4$OMe gave [4,3-*MeO*(*AcNH*)-C_6H_3]$_2$*CHCCl*$_3$, m. 213.14° (from EtOH), contains 1 EtOH; *free amine*, m. 60°; *di-HCl salt*. Similarly 7.5 g. *o*-AcNHC$_6$H$_4$OH gave 10.2 g. [4,3-*HO*(*AcNH*)-C_6H_3]$_2$*CHCCl*$_3$, m. 226.7° (from 60% EtOH); *free amine*, solid (from C_6H_6), decomp. 152°. The *p*-isomer gave only 6-*acetamido*-2,4-*bis*(*trichloromethyl*)-1,3-*benzodioxane*, m. 205° (cf. Chattaway, C. I. 21, 233). Boiling 2 g. I 12 hrs. with 10 ml. AcOH and 1 g. 92% H_2SO_4 gave 1.6 g. poorly sol. *di-HCl salt* of the *di-HO analog*, m. 242°; *free amine*, m. 175° (from C_6H_6), is poorly stable. A reaction analogous to the prepn. of I with 7.5 g. *m*-AcNHC$_6$H$_4$OH gave 4 g. [4,2-*HO*(*AcNH*)C_6H_3]$_2$*CHCCl*$_3$, m. 186° (from C_6H_6-EtOH), which could not be deacetylated without decompn., but treatment with EtONa and MeI in EtOH gave the *di-MeO analog*, m. 129° (from dil. EtOH). Analogously, *m*-MeOC$_6$H$_4$NHAc gave 2,4-*AcNH*(*MeO*)-C_6H_3*CH*(*OH*)*CCl*$_3$, m. 179° (from EtOH); hydrolysis with 17% HCl gave the *HCl salt* of the *free amine*. G. M. K.

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

Chem A

10

Synthesis and structure of some colchicine derivatives

M. N. Shchukina, G. M. Borodina, and Yu. N. Sheinker (S. Ordzhonikidze All-Union Chem. Pharm. Inst., Moscow). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 735-9(1951); cf. Santavy, *C.A.* 42, 5891a.—Heating 1 g. *colchicine* (I) and 1.5 ml. 5% alc. NH_3 4 hrs. to 100° gave 0.64 g. crystals, identified as *aminocolchiceine*, m. 260-2°, $[\alpha]_D^{20}$ −144.31° ($CHCl_3$), giving in aq. soln. pH 7.8; the product contains 0.5 mole EtOH of crystn. Letting I stand with EtOH-$MeNH_2$ 3 days at room temp. in a sealed tube gave the *methylamino analog*, m. 172-8°, while heating I with Et_2NH 2.5-3 hrs. at 100° gave the *diethylamino analog*, m. 208-9°, $[\alpha]$ 521.62° ($CHCl_3$). $PhCH_2NH_2$ (6 hrs. at 100° in EtOH soln.) gave the *benzylamino analog*, m. 120-4°, $[\alpha]$ −123.18° ($CHCl_3$). In all cases the amino group replaces the MeO group in the C ring. Treatment of the amino deriv. with EtO_2CCl in dioxane in the presence of dry K_2CO_3 (8 hrs. at 85°) gave the *carbethoxyamino analog*, m. 112-20°, $[\alpha]$ −127.09° ($CHCl_3$). Heating 1 g. I with 0.1 g. Na in 5 ml. abs. EtOH 4 hrs. at 100° in a sealed tube, followed by treatment of the evapn. residue with MeI (4 hrs. at 100°) gave 0.5 g. *Me colchicinate*, m. 254-5° (from EtOH), $[\alpha]$ −132.71°, showing the aromatization of the C ring. Heating I with MeONa-MeOH 4 hrs. at 100° and acidification gave *colchicinic acid*, m. 257-8°. A similar reaction, followed by treatment of the product with EtI at 100°, gave 20% *Et colchicinate* (purified by chromatography on Al_2O_3 in $CHCl_3$-Et_2O, elution by EtOH), m. 217°, $[\alpha]$ −133.7° ($CHCl_3$), shows an absorption max. near 2800 A. while the amino deriv. shows max. at 4050, 3600, and 2400 A. and methylamino deriv. gives max. at similar positions (last max. near 2500 A.). G. M. Kosolapoff

1957

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

CA

Synthesis and structure of some colchicine derivatives. M. N. Shchukina, G. M. Boroslina, and Yu. N. Shenker. *J. Gen. Chem. U.S.S.R.* 21, 809-13(1951)(Engl. translation). See C.A. 45, 9549f. R. M. S.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. N.

USSR/Chemistry - Antitubercular Drugs Jan 52

"Synthesis of Certain Aminoguanidine Derivatives," M. N. Shchukina, Ye. Ye. Mikhlina, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonokidze

"Zhur Obshch Khim" Vol XXII, No 1, pp 132-135

Prepd a number of substituted benzalguanyls and their N^4-alkyl derivs. Found from tests at Div of Chem Therapy, All-Union Sci Res Chem-Phar Inst that these compds possess tuberculostatic activity but are toxic.

207T25

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

USSR/Chemistry - Quinoline Derivatives Jul 52
Medicine - Antibacterial Drugs

"The Derivatives of 8-Hydroxyquinoline and Their Antibacterial Action. I. 8-Aryloxyquinolines and 8-Alkoxyquinolines," M. N. Chchukina, N. V. Savitskaya, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol 22, No 7, pp 1218-1224

The following compds were synthesized: 8-phenoxyquinoline, with its p-amino-, p-acetamino-, and p-hydroxyderivatives, and 5-phenoxy-8-methoxyquinoline; a series of 8-alkoxyquinolines

229T44

with the alkyls C_3 - C_{12}; 5-nitro-8-butoxyquinoline, 5-acetamino-8-butoxyquinoline, 5,7-dibromo-8-butoxyquinoline, and 5,7-dichloro- and 5,7-dibromo-8-octyloxyquinolines. All these compds are characterized by antibacterial and anti-enzyme (anti-indophenoloxidase) action.

229T44

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. N.

USSR/Chemistry - Quinoline Derivatives Jul 52
Medicine - Antibacterial Drugs

"The Derivatives of 8-Hydroxyquinoline and Their Antibacterial Action. II. N-Oxides of 8-Hydroxyquinoline and Its Ethers," M. N. Shchukina, N. V. Savitskaya, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol 22, No 7, pp 1224-1228

The N-oxides of 8-hydroxyquinoline and 5-chloro-8-hydroxyquinoline, and the N-oxides of 8-alkoxyquinolines with the alkyl radicals C_1 -C_{11} were

229T45

obtained. States that these compds, despite their inability to form complexes, nevertheless show definite antibacterial action.

229T45

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

MAYMIND, V.I.; SHCHUKINA, M.N.; ZHUKOVA, T.F.

Microsynthesis of labelled S^{35}-methionine. Zhur. Obshchey Khim. 22, No. 7 1234-6 '52. (MLRA 5:8)
(CA 47 no.13:6346 '53)

1. S. Ordzhonikidze All-Union Chem. Pharm. Inst., Moscow.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

Chem. 6 (3)

Biological Abst.
Vol. 28 No. 5
Sec. 10. 1954
Medicinal Chemistry

The derivatives of 8-hydroxyquinoline and their antibacterial activity. I. 8-Aryloxy- and 8-alkoxyquinolines. M. N. Shchukina and N. V. Savitskaya (S. Ordzhonikidze All-Union Research Inst. Pharm. Chem., Moscow). *Zh. Obshch. Khim. (J. Gen. Chem. U.S.S.R.)* 22, 1263-8(1952)(Engl. translation).—*C.A.* 47, 7501*f*. II. The *N*-oxides of 8-hydroxyquinoline and its ethers. *Ibid.* 1269-72.—See *C.A.* 47 7501*i*.

H. L. H.

AF 7-13-54

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

USSR/Chemistry - Pharmaceuticals Sep 52

232T31

"Some Syntheses in the Series of 4-Acetaminobenzaldehydes," M. N. Shchukina, G. M. Borodina, Ye. D. Sazonova, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol 22, No 9, pp 1659-1663

A series of aromatic aldehydes contg substituents in the 2 and 3 positions as well as an acetamino group in the 4 position were obtained. The thiosemicarbazones of these aldehydes were prepd. By treating 4-amino-2- and 3-methoxybenzaldehydes

232T31

with succinic anhydride or benzoyl chloride, the corresponding acylated compds were obtained. The prepn of 4-acetamino-2-methoxycinnamic acid, 4-acetamino-2-methoxyhydroxycinnamic acid, and 4-amino-2-oxyhydrocinnamic acid is described.

232T31

SHCHUKINA, M. N.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

USSR/Chemistry - Pharmaceuticals Sep 52

"The Synthesis of Some p-Alkylsulfonylbenzaldehydes," M. N. Shchukina, T. P. Sycheva, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol 22, No 9, pp 1663-1667

232T32

A method of prepg p-alkylsulfonylbenzaldehydes was developed which consists of the oxidation of alkyl-p-tolylsulfones with chromic anhydride in the presence of glacial acetic acid, acetic anhydride, and concentrated sulfuric acid. A number of

232T32

p-alkylsulfonylbenzaldehydes and their derivs were obtained.

232T32

M. N. Shchukina,

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. N.

CATALYSTS

Chemical Abst.
Vol. 48 No.8
Apr. 25, 1954
Organic Chemistry

No 9

(3) fuel

Some syntheses in the p-acetamidobenzaldehyde series. M. N. Shchukina, G. M. Borodina, and E. D. Sazonova. J. Gen. Chem. (U.S.S.R.) 22, 1701-4(1952)(Engl. translation).—See C.A. 47, 9298a. H. L. H.

8-30-54

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M.N.; GORTINSKAYA, T.V.

Aromatic disulfides and mercaptans. I. 2-Mercapto-4-nitrobenzoic acid and its transformations. Zhur. Obshchey Khim. 22,1855-61 '52. (CA 47 no.13:6366 '53)

(MLRA 5:11)

1. S. Ordzhonikidze All-Union Chem. Pharm. Inst., Moscow.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

1. SHCHUKINA, M. N., SYCHEVA, T. P.

2. USSR (600)

4. Phenylalanine

7. Synthesis of some analogs of phenylalanine. Zhur. ob. khim., 22, No. 10, 1952

p 1879

9. Monthly List of Russian Accessions, Library of Congress, February 1953. Unclassified.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M.N.

3

Chemical Abst.
Vol. 48 No. 8
Apr. 25, 1954
Organic Chemistry

Aromatic disulfides and mercaptans. I. 2-Mercapto-4-nitrobenzoic acid and its transformations. M. N. Shchukina and T. V. Gortinskaya. *J. Gen. Chem.* (U.S.S.R.) 23, 1895-1900(1953)(Engl. translation).—See *C.A.* 47, 6366a. H. L. H.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. N.

4

2

Synthesis of some analogs of phenylalanine. M. N. Shchukina and T. P. Sycheva. *J. Gen. Chem.* (U.S.S.R.) 22, 1919-22(1952)(Engl. translation).—See *C.A.* 47, 5375*d*. H. L. H.

Chemical Abst.
Vol. 48 No. 8
Apr. 25, 1954
Organic Chemistry

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

USSR/Chemistry - Synthetic Drugs · Nov 52

"Synthesis of Certain Derivatives of p-Aminosalicylic Acid," E. S. Golombik and M. N. Shchukina, All-Union Sci-Res Chem-Pharm Inst imeni S. Ordzhonikidze

"Zhur Obshch Khim" Vol 22, No 11, pp 2014-2019

The purpose of this work was to find anti-tuberculosis drugs which, unlike p-aminosalicylic acid are not eliminated too quickly from the organism. The derivs of p-aminosalicylic acid gave promise of being eliminated more slowly by the organism.

238T33

Therefore, a series of derivs of this acid was synthesized: esters, N-carbalkoxy derivs, N-acyl-p-aminosalicylic acids, /bis-(4-carbethoxy-3-oxyphenyl)/-urea and /bis-(4-carbethoxy-3-oxyphenylamino)/-methane.

238T33

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

K. N.

238T34

USSR/Chemistry - Synthetic Drugs Nov 52

"Synthesis of Homologues of p-Aminosalicylic Acid," M. N. Shchukina, Yu. V. Markova and A. M. Pozharskaya, All-Union Sci-Res Chem-Pharm Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol 22, No 11, pp 2019-2021

Synthesized homologues of p-aminosalicyclic acid in order to explain the effect of a side chain on the anti-tubercular activity. Through a series of intermediate products, prepared 2-hydroxy-4-amino-5-methylbenzoic acid from 2-amino-4-nitro-toluene, and 2-hydroxy-4-amino-6-methylbenzoic acid from 3-hydroxy-5-nitro-toluene.

238T34

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M.N.

2 Chem

Synthesis of some derivatives of p-aminosalicylic acid. E. S. Golombik and M. N. Shchukina. *J. Gen. Chem. U.S.S.R.* 22, 2069-73(1952)(Engl. translation).—See *C.A.* 47, 8686f. H. L. ~~H.~~

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

5

Chem

Chemical Abst.
Vol. 48 No. 9
May 10, 1954
Organic Chemistry

Synthesis of homologs of p-aminosalicylic acid. M. N. Shchukina, Yu. V. Markova, and A. M. Pozharskaya. J. Gen. Chem. U.S.S.R. 22, 2075-6(1952)(Engl. translation).—See C.A. 47, 9299i. H. L. H.

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

USSR/Chemistry - Pharmaceuticals 11 Jun 52
Medicine - Tuberculosis, Chemotherapy

"Isonicotinoyl Hydrazones and Their Antitubercular Activity," M. N. Shchukina, G. N. Pershin, O.O. Makeyeva, Ye. D. Sazonova, Ye. S. Nikitskaya, A. D. Yanina, A. I. Yakovleva, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze

"Dok Ak Nauk SSSR" Vol LXXXIV, No 5, pp 981 - 984

Isonicotinoyl hydrazide has an antitubercular action, but its therapeutic index is low. A series of substituted isonicotinoyl hydrazide derivs were prepd and their bacteriostatic action on tuberculosis

223T17

bacilli in vitro tested. It was shown that substituted isonicotinoyl hydrazones have a higher antitubercular activity than para-aminosalicylic acid and streptomycin and are better tolerated by exptl animals than the hydrazide of isonicotinic acid. This made it possible to select from them substances for clinical study on tubercular humans. A substance, called "Phtivacide" by the authors, was forwarded for clinical study, which is progressing successfully. Presented by Acad A. N. Nesmeyanov 9 Apr 52.

223T17

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. N. and MAKEYEVA, O. O.

"The Antituberculosis Activities of an Amino Derivative and a Hydroxy Derivative of Diphenyltrichlorethane," Zhurnal Mikrobiologii, Epidemiologii i Immunobiologii, No 1, 1953.

VNIKhFI, All-Union Scientific Research Chemicopharmaceutical Institute imeni S. Ordzhonikidze

APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4"

"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M. N.

Alkylation of the ethyl ester of β-hydroxycapric acid. G. M. Borodina and M. N. Shchukina (S. Ordzhonikidze All-Union Sci. Research Chem. Pharm. Inst., Moscow). *Sbornik Statei Obshchei Khim., Akad. Nauk S.S.S.R.* 1, 382-4(1953).—Addn. of 42 g. $C_7H_{15}CHO$ and 60 g. $BrCH_2CO_2Et$ in 60 ml. C_6H_6 to 23.5 g. activated Zn dust under 25 ml. Et_2O, followed by brief warming, started a vigorous reaction which required external cooling even when the addn. extended over 1-2 hrs. The mixt. heated 1.5 hrs. longer, treated with 10% H_2SO_4 and the org. layer sepd., washed and distd. yielded 48% $C_7H_{15}CH(OH)CH_2CO_2Et$ (I), b_{10} 130-5°, d_{20} 0.9354, n_D^{20} 1.4365. I (10.5 g.) and 24 g. EtI in 20 ml. EtOH were treated with 18 g. dry Ag_2O and the mixt. stirred at reflux 20 hrs., filtered and the filtrate distd. yielding 5 g. I and 30% $C_7H_{15}CH(OEt)CH_2CO_2Et$, b_{10} 93°, d_{20} 0.8802, n_D^{20} 1.420. Similarly were obtained the following *alkoxy analogs* of I: *BuO*, b_{10} 104-6°, d_{20} 0.8721, n_D^{20} 1.4230; $C_6H_{13}O$, b_{10} 110-14°, d_{20} 0.8714, n_D^{20} 1.4225; $C_8H_{17}O$, b_{10} 155-7°, d_{20} 0.8713, n_D^{20} 1.4313. Hydrolysis of the corresponding esters with 40% KOH 12 hrs. at room temp. gave the following free acids: $C_7H_{15}CH(OEt)CH_2CO_2H$, b_{10} 120-5°, m. 13-14°; *BuO analog*, b_9 119-21°, f.p. about 5°; $C_8H_{17}O$ *analog*, b_{10} 113-16°, f.p. 0-2°. The yields of both the esters and the acids were rather low. G. M. K.

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SHCHUKINA, M.N.

Synthesis of N-substituted derivatives of p-sulfamoylbenzaldehyde and its thiosemicarbazones. T. P. Sycheva and M. N. Shchukina (S. Ordzhonikidze All-Union Chem. Pharm. Inst., Moscow). *Sbornik Statei Obshchei Khim., Akad. Nauk S.S.S.R.* 1, 527-32(1953).—To 17.1 g. p-$MeC_6H_4SO_2Cl$ in 142 ml. AcOH and 142 ml. Ac_2O was added, with cooling to 0°, 21.5 g. concd. H_2SO_4, followed by 25 g. powd. CrO_3 at 9-12°, and the mixt. stirred 0.5 hr., and quenched in ice to yield 25.3 g. solid product which was extd. with cold $(CH_2Cl)_2$ leaving 3.4 g. p-$ClSO_2C_6H_4CO_2H$, m. 225-6° (decompn.). The ext. on evapn. gave 50% p-$ClSO_2C_6H_4CH(OAc)_2$ (I), m. 111-13° (from Me_2CO-petr. ether). To 30 ml. Me_2CO, 15 ml. H_2O, and 5 ml. 25% NH_4OH was added with cooling 5 g. I in Me_2CO at 5°; after evapn. of Me_2CO there was obtained 83.3% p-$H_2NSO_2C_6H_4$-$CH(OAc)_2$, m. 143-5° (from EtOH); refluxing this with aq. alc. H_2SO_4 1 hr. gave 81.6% p-$H_2NSO_2C_6H_4CHO$, m. 123-4°, which with thiosemicarbazide in aq. EtOH gave the *thiosemicarbazone*, m. 237°. Refluxing I with 1:10 HCl gave on evapn. and treatment with NaCl a ppt. of $NaO_2SC_6H_4CHO$; *thiosemicarbazone*, $C_8H_9O_2N_4S_2$, decomp. 270°, which, when neutralized with NaOH, gave the *corresponding Na salt*, a solid. Similarly I gave the following p-$RSO_2C_6H_4CH(OAc)_2$ (R given): Et_2N, 91%, m. 86-7.5°; Pr_2N, 96%, m. 80-2°; $BuNH$, 98%, m. 105-7°; $C_6H_{11}NH$, 97%, m. 50-3°; *morpholino*, 88%, m. 133-5°; p-$O_2NC_6H_4NH$, 16%, m. 161-2°; p-$MeOC_6H_4NH$, 93%, m. 126-8°; *2-pyridylamino*, 67%, m. 181-3°; *4-carboxy-3-hydroxyphenylamino monohydrate*, m. 164-6°. These were hydrolyzed as above to the p-$RSO_2C_6H_4CHO$ (R given): Et_2N, 95%, m. 77-9°; $BuNH$, 97%, m. 59-63°; $C_6H_{11}NH$, no yield given, m. 74-6°; *morpholino*, 95%, m. 142-4°; p-$MeOC_6H_4NH$, 90%, m. 103-5°; p-$O_2NC_6H_4NH$, 89%, m. 133-5°; *2-pyridylamino*, 83%, m. 176-8° (decompn.); *3-hydroxy-4-carboxyphenylamino*, 33%, m. 215-16°. The thiosemicarbazones, p-$RSO_2C_6H_4CH{:}NNHC(S)NH_2$, were made from the corresponding aldehyde (R given): Et_2N, m. 206-8° (decompn.); Pr_2N, m. 195-6°; $(HOCH_2CH_2)_2N$, m. 202-3°; $(C_6H_{11})_2N$, m. 169-70°; $BuNH$, m. 207-8°; $C_6H_{11}NH$, m. 165-6°; *morpholino*, m. 230°; p-$MeOC_6H_4NH$, decomp. 212°; p-$O_2NC_6H_4NH$, decomp. 213-15°; *2-pyridylamino*, m. 217°; *4-carboxy-3-hydroxyphenylamino*, decomp. 233°.

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SHCHUKINA, M.N.

Synthesis of N-substituted 4-sulfamoylbenzoic acids. I. T. P. Sycheva, N. V. Savitskaya, and M. N. Shchukina (S. Ordzhonikidze All-Union Chem.-Pharm. Inst., Moscow). *Sbornik Statei Obshchei Khim., Akad. Nauk S.S.S.R.* 1, 568-71(1953).—Heating 77.5 g. *p*-$MeC_6H_4SO_2NH_2$, 45 g. 40% H_2OH, 100 g. PrBr and 450 ml. EtOH in an autoclave 6 hrs. at 100-10°, then addn. of 25 ml. 40% NaOH and heating 2 hrs. longer, gave, after evapn., extn. with Et_2O, and distn. of the ext. 72.5% *p*-$MeC_6H_4SO_2NPr_2$, b_{1-4} 156-8°; the reaction of RSO_2Cl with Pr_2NH in alc. KOH gave 56% product. To 50 ml. *N* NaOH and 2.5 g. Pr_2NH was added with cooling 5 g. *p*-$HO_2CC_6H_4SO_2Cl$ in Me_2CO, the mixt. stirred 1 hr., the solvent evapd., the residue dild. with H_2O and acidified to obtain 50% *p*-$HO_2CC_6H_4SO_2NPr_2$, m. 196-7° (from EtOH). Similarly was prepd. 80% *p*-$HO_2CC_6H_4SO_2NEt_2$, m. 192-4° (from EtOH), and *p*-$HO_2CC_6H_4SO_2NBu_2$, m. 166-7°. Addn. of 7 g. *p*-$HO_2CC_6H_4SO_2Cl$ in Me_2CO to 10 g. $(HOCH_2CH_2)_2NH$ in 50 ml. H_2O at 0-5° yielded 4 g. *p*-$HO_2CC_6H_4SO_2N(CH_2CH_2OH)_2$, m. 217-20° (from 80% EtOH); similarly (in the presence of NaOH) was prepd. 85.5% *p*-$HO_2CC_6H_4SO_2N(CH_2CH_2)_2O$, m. 260-1° (from AcOH), which, refluxed in EtOH in the presence of dry HCl 13 hrs., gave the *Et ester*, m. 92-4° (from dil. EtOH).

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SHCHUKINA, M.N.

Synthesis of *N*-substituted *p*-aminobenzalthiosemicarbazones. M. N. Shchukina and E. D. Sazonova (S. Ordzhonikidze Mikhiyan Chem.-Pharm. Inst., Moscow). *Sbornik Statei Obshchei Khim.* 2, 1031–4(1953).—To a refluxing soln. of 100 g. *p*-$O_2NC_6H_4Me$ in 100 ml. EtOH was added in 1.5 hrs. a hot soln. of 60 g. S in 825 ml. 12% NaOH, the mixt. stirred and heated 3 hrs. longer, cooled to 30°, the upper layer sepd. and treated with a hot soln. of 42 g. thiosemicarbazide in 250 ml. H_2O, heated 15 min., cooled to 20° and neutralized with 40 ml. 80% AcOH, yielding 89.3% *p-aminobenzalthiosemicarbazone* (I), m. 197–8° (from 2% HCl). This treated with Ac_2O in Me_2CO 3–4 hrs. gave 92.1% *Ac deriv.*, m. 224–6°. To 1.4 g. $ClCH_2CO_2H$ in 1.5 g. $NaHCO_3$ and 30 ml. H_2O was added 1.94 g. I and the mixt. refluxed 2.5 hrs. and cooled, yielding 94% *p-glycylaminobenzalthiosemicarbazone*, m. 270–1° (from 50% AcOH); this (3.88 g.) in 100 ml. EtOH was treated with 3.8 g. glucose in 25 ml. H_2O and 6.15 g. 40% $NaHSO_3$ and refluxed 4 hrs.; on cooling there is obtained a ppt. of 40% colorless *glucose bisulfite deriv.*, $C_{14}H_{21}N_4O_8S_2Na$, m. 221° (from EtOH). Heating 19.1 g. I with 15 g. formaldehyde $NaHSO_3$ complex and 0.5 ml. 40% $NaHSO_3$ (solvent not specified) 1 hr. gave 70.4% I *formaldehyde bisulfite complex*, $C_8H_{11}N_4O_4S_2Na.H_2O$, decomp. 225° (from H_2O). Heating 3 g. I with 2.2 g. $Et_2NCH_2CH_2Cl$ 3 hrs. at 80° gave 3.2 g. *p-2-diethylaminoethylaminobenzalthiosemicarbazone*, yellow, m. 186–7° (from H_2O). Addn. of 1.1 g. $(CH_3CO)_2O$ to 1.94 g. I in Me_2CO gave a ppt. of 85% *p-succinylaminobenzalthiosemicarbazone*, m. 199–200° (from EtOH). I with BzCl similarly gave the *p-benzamido deriv.*, 83.9%, decomp. 267–8° (from EtOH). Nicotinoyl chloride in C_5H_5N-pyridine gave with *p*-

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$H_2NC_6H_4CHO$, colorless *p-nitrosoaminobenzaldehyde*, m. 174–5° (from H_2O); *thiosemicarbazone*, decomp. 2[illegible]–1° (from EtOH). Heating 5 g. *p*-aminosalicylic acid with 25 ml. Ac_2O 2 hrs. gave 78% *2-acetoxy-4-acetamidobenzoic acid*, m. 189–90° (from EtOH). This (3 g.) heated 3 hrs. at 60° with 5 g. $SOCl_2$, evapd. *in vacuo*, and the residual acyl chloride treated with 1.5 g. *p*-$H_2NC_6H_4CHO$ in C_6H_6 and 2 ml. pyridine, and refluxed 3 hrs. gave 0.5 g. *4-(2-hydroxy-4-acetamidobenzoyl)aminobenzaldehyde*, m. 214–15° (from H_2O); *thiosemicarbazone*, decomp. 235–6° (from EtOH).

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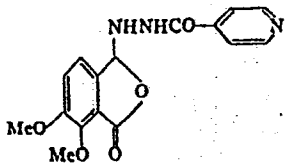
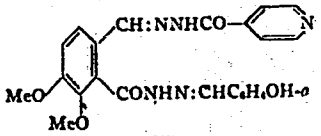
SHCHUKINA, M.N.

Syntheses in the series of derivatives of isonicotinic acid hydrazide. M. N. Shchukina and E. D. Saxonova (S. Ordzhonikidze All-Union Chem. Pharm. Inst., Moscow). *Zhur. Obshchei Khim.* 23, 687–90(1963); cf. *C.A.* 46, 10431h.—

Opianic acid [2,3,4-$HO_2C(MeO)_2C_6H_2CHO$] (2.1 g.) in 40 ml. hot H_2O added a hot soln. of 1.37 g. isonicotinic hydrazide (I) gave on cooling 98% *isonicotinoylhydrazone* (II), m. 205–6° (*monohydrate*, loses H_2O at 160°); *Na salt*, crystals (from H_2O), m. 239–40°; diethanolamine salt, m. 196–7° (from EtOH); *Et_3NH salt*, m. 176–7° (from EtOH). Aq. solns. of these salts become cloudy on standing and deposit ppts. of the salts. Addn. of 1.37 g. I in 10 ml. H_2O to 2.1 g. Et pseudoopianate [*6,2,3-$EtO_2C(MeO)_2C_6H_2CHO$*] gave a cyclic compd. (IIa), m. 207–8°. Heating II in EtOH gave III. IIa and III appear to be the same compd., but a mixed m.p. depresses to 190–8°. Similarly was prepd.



(III)



(IV)

2-carboxy-3,4-dimethoxybenzaldehyde nicotinoylhydrazone (IIb), (97%), m. 214–16° (*monohydrate*, loses H_2O at 150°). II (3.2 g.) heated 3 hrs. in 50 ml. abs. EtOH satd. with HCl gave 2.5 g. *Et ester* of II, m. 213–14° (from EtOH), which with $N_2H_4.H_2O$ rapidly gave 87% *2-NH_2NHCO analog*, m. 152–3° (from H_2O). This (1.5 g.) in EtOH with 0.6 g. *o*-HOC_6H_4CHO in 10 ml. hot EtOH gave 1.9 g. *2-(o-HO-C_6H_4CH:$NNHCO$) analog* (IV), m. 210–11° (from EtOH). Heating 10 g. 4-chloropyazine [cf. Gabriel, *Ber.* 36, 3378 (1903)] and 20 g. $N_2H_4.H_2O$ to 30° led to a spontaneous reaction and on cooling the mixt. yielded *4-hydrazino-5,6-dimethoxyphthalazine*, m. 75–6°; *HCl salt*, m. 164–5° (from H_2O). Heating 2.25 g. 4-chloropyrazine with 1.37 g. I in EtOH 1 hr. gave 90% *5,6-dimethoxy-4-(isonicotinoylhydrazino)phthalazine*, decompd. 234–5° (from EtOH). Similarly was prepd. the *nicotinoylhydrazino isomer* (90%), m. 207–8°. The latter and IIa are less active against the tuberculosis bacteria than the corresponding isonicotinic derivs. Introduction of a 2nd :CNHN:C group into the isonicotinoylhydrazones reduces their activity. G. M. K.

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SHCHUKINA, M.N.

USSR.

Synthesis in the series of derivatives of isonicotinic acid hydrazide. M. N. Shchukina and E. D. Sazonova, J. Gen. Chem. U.S.S.R. 23, 715-17(1953)(Engl. translation). See C.A. 48, 7602i. H. L. H.

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SHCHUKINA, M.N.

PERSHIN, G.N., laureat Stalinskoy premii, professor, redaktor; SHCHUKINA, M.N., professor, redaktor; NATRADZE, A.G., otvetstvennyy sekretary; SERGIYEVSKAYA, S.I., professor, chlen redaktsionnoy kollegii; MAGIDSON, O.Yu., professor, laureat Stalinskoy premii, chlen redaktsionnoy kollegii; UTKIN, L.M., professor, chlen redaktsionnoy kollegii; MASHKOVSKIY, M.D., professor, chlen redaktsionnoy kollegii; KARAKHANYAN, O.I., redaktor; GLUKHOYEDOVA, G.A., tekhnicheskiy redaktor.

[Synthomycin] Sintomitsin. Otvet. red. G.N.Pershin. Moskva, Gos. izd-vo med. lit-ry. 1954. 194 p. (MLRA 7:8)

1. Moscow. Nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut.
(Chloromycetin)

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SHCHUKINA, M.N.; SAZANOVA, Ye.D.; PERSHIN, G.N.; Makeyeva, O.O.

Aromatic isonicotinylhydrazones; a new class of drugs in the treatment of tuberculosis. Probl.tub. no.2:44-50 Mr-Ap '54. (MLRA 7:5)

1. Iz Vsesoyuznogo nauchno-issledovatel'skogo khimiko-farmatsevticheskogo instituta.
(TUBERCULOSIS, experimental,
*eff. of isonicotinaldehyde thiosemicarbazone)
(ALDEHYDES, effects,
*isonicotinaldehyde thiosemicarbazone, on exper. tuberc.)
(THIOSEMICARBAZONES, effects,
*isonicotinaldehyde thiosemicarbazone, on exper. tuberc.)

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Shchukina, M. N.

USSR/ Chemistry - Pharmaceutics

Card 1/1 Pub. 151 - 28/36

Authors : Savitskaya, N. V., and Shchukina, M. N.

Title : Synthesis of phenothiazine-1-carboxylic acid derivatives

Periodical : Zhur. ob. khim. 24/1, 152-156, Jan 1954

Abstract : Various amides and hydrazides of phenothiazine-1-carboxylic acid and some of their derivatives were synthesized and their anti-bacterial (anti-tubercular) effects were investigated. The effect of a thionyl chloride surplus, phosphorous oxychloride and phosphorous pentachloride on phenothiazine-1-carboxylix acid, is explained. The synthesis of acid chloride of trichlorophenothiazine-1-carboxylic acid, according to Curtius, results in the formation of an imidazole cycle. Six references: 3-USA; 2-USSR and 1-German (1944-1953).

Institution : All-Union Scientific Research Chemical-Pharmaceutical Institute

Submitted : July 14, 1953

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SHCHUKINA, M. N.

USSR/Chemistry

Card 1/1

Authors : Shchukina, M. N.; and Yuan'-Chen-e

Title : Synthesis of sulfanilamide derivatives in the lysine series.

Periodical : Zhur. Ob. Khim, 24, Ed. 4, 687 - 692, April 1954

Abstract : Lysine is one of the irreplaceable amino acids of great importance for the metabolism and growth processes in the organism. Its sulfanilil substitutes are of interest in testing its antibacterial effect. The authors synthesized Nα - and Nε -sulfanilil substitutes of d, l-lysine through its copper complex. The authors obtained Nα - Nε - disulfanilil substitutes of d, l-lysine and corresponding ester, hydrazide and hydrazones. Nine references; 1 USSR since 1952; 1 German 1909; 7 English since 1934. Chemical formulas.

Institution : The S. Ordzhonikidze All-Union Scientific-Research Chemical-Pharmaceutical Institute.

Submitted : November 30, 1953

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"APPROVED FOR RELEASE: 08/23/2000 CIA-RDP86-00513R001548920017-4

SHCHUKINA, M.N.

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USSR. Synthesis of sulfanilamide derivatives in the lysine series. M. N. Shchukina and Yuan-Cheu-e. J. Gen. Chem. U.S.S.R. 24, 896-9(1954)(Engl. translation).—See C.A. 49, 6153e. H. L. H.

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Shchukina, M. N.

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Synthesis of derivatives of *p*-hydroxybenzenesulfonic acid. N. V. Savitskaya and M. N. Shchukina. *J. Gen. Chem. U.S.S.R.* 24, 2017-20(1954)(Engl. Translation).—See *C.A.* 49, 14665*b*. B. M. R.

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SHCHUKINA, M. N.

4

Synthesis of derivatives of *p*-hydroxybenzenesulfonic acid.

N. V. Cavitskaya and M. N. Shchukina (S. Ordzhonikidze All-Union Sci. Research ~~Chem.-Pharm.~~ Inst., Moscow).

Zhur. Obshchei Khim. 24, 2052-5(1954). —$N_2H_4.H_2O$ (5 g.) in 5 ml. H_2O treated slowly with 10 g. *p*-$BzOC_6H_4SO_2Cl$ and cooled yielded 71% *p*-$HOC_6H_4SO_2NHNH_2$, decomp. 165-6°, (from H_2O). This with *p*-HOC_6H_4CHO in hot H_2O gave *p*-(*p*-$HOC_6H_4SO_2NHN:CH)C_6H_4OH$, m. 148-9° (from aq. EtOH). *p*-$AcNHC_6H_4CHO$ similarly gave *p*-(*p*-$HOC_6H_4SO_2NHN:CH)C_6H_4NHAc$, decomp. 184-5° (from aq. EtOH), while *p*-$Me_2NC_6H_4CHO$ gave *p*-(*p*-$HOC_6H_4SO_2NH...CH)C_6H_4NMe_2$, decomp. 157-8° (from 25% AcOH). $N_2H_4.H_2O$ and *p*-$EtOC_6H_4SO_2Cl$ similarly gave 76% *p*-$EtOC_6H_4SO_2NHNH_2$, m. 111° (decompn.; from EtOH), converted to the following *p*-$EtOC_6H_4SO_2NHN:CHR$ (R given): *p*-$AcNHC_6H_4$, decomp. 186-6.5° (from EtOH); *p*-$Me_2NC_6H_4$, decomp. 170-1° (from EtOH); 3,4-Me(HO)C_6H_3, decomp. 128-30°. The reaction of 16 g. *p*-$AcNHC_6H_4NH_2$ (16 g.) with 24 g. *p*-$BzOC_6H_4SO_2Cl$ in H_2O in the presence of 17 g. Na_2CO_3 (completed in 1 hr. at 40°) gave *p*-(*p*-$EtOC_6H_4SO_2NH)C_6H_4NH_2$, m. 155-6°, after hydrolysis of the intermediate by refluxing 2 hrs. with 100 ml. 20% NaOH. Similar treatment of *p*-$BzOC_6H_4SO_2Cl$ gave *p*-(*p*-$HOC_6H_4SO_2NH)C_6H_4NH_2$, m. 200-1° (from H_2O). Heating 15 g. *p*-$C_6H_4(NH_2)_2$ with 9 g. *p*-$EtOC_6H_4SO_2H$ in H_2O 2.5 hrs. at 45° gave *p*-(*p*-$EtOC_6H_4SO_2NH)_2C_6H_4$, decomp. 246° (from AcOH). $AcNHCH_2CH_2NH_2$ with *p*-$BzOC_6H_4SO_2H$ gave an intermediate product which, refluxed with 20% HCl 26 hrs., was hydrolyzed to *p*-$HOC_6H_4SO_2NHCH_2CH_2NH_2$, decomp. 240-3° (from concd. HCl); *p*-$EtOC_6H_4SO_2Cl$ in the above reaction gave, after hydrolysis of the intermediate 26 hrs. with 20% HCl, *p*-$EtOC_6H_4SO_2NHCH_2CH_2NH_2$, decomp. 236° (from concd. HCl). None of the products had a notable antitubercular activity.

G. M. Kosolapoff

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SHCHUKINA, M.N.

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A new method of synthesis of *p*-aminobenzoic acid labeled with carbon-14 and the preparation of labeled anesthetics: anesthesine, Novocaine, and cocaine. Yu. V. Markova, L. N. Zenkova, and M. N. Shchukina (S. Ordzhonikidze All-Union Sci. Research Chem.-Pharm. Inst., Moscow). *Zhur. Obshchei Khim.* 25, 1383–7(1955).—*p*-$ClC_6H_4C^{14}O_2H$ (I) was prepd. in 75% yield by the reaction of BuLi with *p*-BrC_6H_4Cl, followed by treatment with $C^{14}O_2$ at −70° under N. A detailed description of the app. and the procedure is given. I heated in autoclave to 138–40° 4 hrs. with 28% NH_4OH in the presence of Cu_2Cl_2 gave 70.4% *p*-$H_2NC_6H_4C^{14}O_2H$ (II), m. 185°. Refluxing II.HCl with EtOH and 4–5% dry HCl 4 hrs. gave 73.5% of labeled anesthesine (III), m. 90–1°. III heated with $Et_2NCH_2CH_2OH$ in the presence of $Et_2NCH_2CH_2ONa$ catalyst *in vacuo* to 80–5° yielded 42.5% of labeled Novocaine (HCl salt, m. 156°). Heating $PhC^{14}OCl$ with ecgonine Me ether in C_6H_6 6 hrs. at reflux gave labeled cocaine, isolated as HCl salt, m. 192°. Also in *J. Gen. Chem. U.S.S.R.* 25, 1329–32(1955)(Engl. transaction). G. M. Kosolapoff

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Synthesis of N^{α}-sulfanilamide derivatives of the lysine series. Chen-E Yuan and M. N. Shchukina. *J. Gen. Chem. U.S.S.R.* 25, 1917-21(1955)(Engl. translation).—See *C.A.* 50, 8503c. B. M. R.

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Synthesis of N^{ϵ}-sulfanilamide derivatives of the lysine series. Yuan Chen-e and M. N. Shchukina (S. Ordzhonikidze All-Union Sci. Research Chem. Pharm. Inst., Moscow). *Zhur. Obshchei Khim.* 25, 1972-7(1955).—Beckmann rearrangement of cyclohexanone oxime and direct benzoylation of the crude product gave 73% $BzNH(CH_2)_5CO_2H$, m. 78-80°. Bromination and esterification gave 80% $BzNH(CH_2)_4CHBrCO_2Et$, m. 56-7° (from petr. ether), which (6.84 g.), 6.88 g. sulfanilamide, and 0.3 g. KI in dry EtOH were heated in a sealed tube 14 hrs. at 140-60°, and the product hydrolyzed with 16% KOH 5 hrs. at 85-90° to yield 72% $p\text{-}H_2NSO_2C_6H_4NHCH(CO_2H)(CH_2)_4NH_2$, m. 202° (from H_2O). To 110 ml. 20% NaOH was added at 50° simultaneously 50 g. cyclohexanone and 185 ml. 26% H_2O_2 over 45 min.; after 1.5 hrs. at room temp., extn. with Et_2O, acidification and salting out with $(NH_4)_2SO_4$, there was obtained the crude hydroxy acid, which (45-50 g.) was treated with 140 ml. 48% HBr, 35 ml. H_2SO_4, and 5 ml. 26% H_2O_2; after 2 hrs. at room temp. and 5 hrs. at 100° there was isolated 42% $Br(CH_2)_5CO_2H$, converted to *Et ester*, $b_{1.5}$ 100-

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5°. This with sulfanilamide, in the presence of pyridine and KI in dry EtOH after 21 hrs. at 100° gave 60% *p*-H_2N-$SO_2C_6H_4NH(CH_2)_4CO_2Et$, m. 95–6°; hydrolysis with 10% KOH gave the *free acid*, m. 126–6.5° (from H_2O), which with Br in the presence of PCl_3 at reflux 10 hrs. gave 74% *p*-H_2N-$SO_2C_6H_4NH(CH_2)_4CHBrCO_2H$, m. 150–2°, which was esterified with EtOH and heated with K phthalimide 0.5 hr. at 150°; hydrolysis of this with 10% KOH, followed by 20% HCl gave *p*-$H_2NSO_2C_6H_4NH(CH_2)_4CH(NH_2)CO_2H$, 51%, m. 186–7° (from dil. EtOH). To 6.3 g. $Br(CH_2)_4CO_2H$ in 10 ml. PCl_3 was added 7.5 g. Br and the mixt. after 0.5 hr. at room temp. was stirred 7 hrs. at 70–98°, yielding on diln. with EtOH, 76% $Br(CH_2)_4CHBrCO_2Et$, $b_{1.4}$ 128–30°. This heated with sulfanilamide and pyridine in the presence of $Cu(OAc)_2$ in dry EtOH 20 hrs. at 160–80° and the crude product hydrolyzed with 10% KOH gave after removal of Cu with H_2S an unstated yield of *bis(N-p-sulfonamidophenyl)-DL-lysine*, m. 227–9° (from aq. EtOH). G. M. K.

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imidazole. I. 4(5)-Phenylimidazolyl-2-mercaptans and sulfides. P. M. Kochergin and M. N. Shchukina (S. Ordzhonikidze All-Union Sci. Research Chem. Pharm. Inst., Moscow). Zhur. Obshchei Khim. 25, 2182–8(1955).—Refluxing 28 g. $BzCH_2NH_2.HCl$ and 17.6 g. KSCN in EtOH 3.5 hrs. gave 23 g. *4(5)-phenyl-2-mercaptoimidazole*, m. 261°; an 80.5% yield is obtained in AcOH soln. The use of p-$O_2NC_6H_4COCH_2NH_2.HCl$ gave 89.4% *4(5)-p-nitrophenyl-2-mercaptoimidazole* (I), does not m. below 320°; in AcOH the yield is 98%. This treated with hot 10% HNO_3 15 min. gave *4(5)-p-nitrophenylimidazole*, m. 225°; *nitrate*; m. 200°. Reduction of I with NH_4-H_2S in aq. EtOH at room temp. gave *4(5)-p-aminophenyl-2-mercaptoimidazole*, m. 237–9° (from EtOH); *HCl salt*, decomp. 260–70°. A soln. of 0.33 g. Na in 18 ml. EtOH was treated with 5 ml. H_2O and 2.52 g. *4(5)-phenyl-2-mercaptoimidazole* (II), followed by 2.13 g. MeI and heating at 45° 4 hrs.; there was obtained 97.5% *4(5)-phenyl-2-methylmercaptoimidazole*, m. 135.5–6.5° (from aq. EtOH); *HCl salt*, m. 189–91°. The use of EtI gave *4(5)-phenyl-2-ethylmercaptoimidazole*, m. 120–30°; *HCl salt*, m. 62–3°, while the use of PrBr gave 98.5% *4(5)-phenyl-2-propylmercaptoimidazole*, m. 78–9°; *HCl salt*, m. 148.5–9°. Similarly was prepd. 99% *4(5)-phenyl-2-butylmercaptoimidazole*, oil; *HCl salt*, m. 172–3°. *4(5)-phenyl-2-isoamylmercaptoimidazole*, m. 81–3° (*HCl salt*, m. 170–70.5°), was prepd. similarly in 90% yield from iso-AmBr, while hexyl bromide gave 91.8% *4(5)-phenyl-2-hexylmercaptoimidazole*, oil; *HCl salt*, m. 130–6.5°. Simi-

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Imidazoles I. 4(5)-Phenyl...

larly, II treated with Na-EtOH, followed by p-$O_2NC_6H_4I$ and refluxed 4 hrs. gave *4(5)-phenyl-2-p-nitrophenylmercaptoimidazole*, decomp. 150–2°; *HCl salt*, m. 242–3.5°. Similarly I and iso-AmBr gave 76.2% *4(5)-p-nitrophenyl-2-isoamylmercaptoimidazole*, oil; *HCl salt*, m. 185–6° (decompn.). II in EtOH-EtONa treated with EtI and refluxed 3 hrs. gave a mixt. from which was obtained a low yield of *1-ethyl-4-phenyl-2-ethylmercaptoimidazole*, oil; *picrate*, m. 184–5°; *free base*, $b_{0.15}$ 157–60°. G. M. Kosolapoff

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Chem Derivatives of diazinecarboxylic acids. T. V. Gortinskaya, K. M. Mutrav'eva, and M. N. Shchukina. *J. Gen. Chem. U.S.S.R.* 25, 2285-8(1955)(English translation).—See *C.A.* 50, 9429*h*. B. M. R. 3

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Imidazole series. II. 4(5)-Phenyl-2-imidazolyl alkyl-(aryl) sulfones and sulfoxides. P. M. Kochergin and M. N. Shchukina. *J. Gen. Chem. U.S.S.R.* 25, 2289–93 (1955)(Engl. translation).—See *C.A.* 50, 9387*i*.

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Shchukina M. N.

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V Derivatives of diazinecarboxylic acids. T. V. Gortinskaya, K. M. Murav'eva, and M. N. Shchukina (S. Ordzhonikidze All-Union Chem. Pharm. Research Inst., Moscow). *Zhur. Obshch. Khim.* 25, 2413-17 (1955).—Brief heating of 25 g. pyrazine-2-carboxylic acid with dry MeOH and 5 ml. H_2SO_4 gave on cooling, neutralization, and extn. with hot EtOAc 70% *Me ester* (I), m. 62°. Similarly was prepd. 70% *Et 3-pyridazone-6-carboxylate*, m. 123-5°, which with $N_2H_4.H_2O$ in EtOH readily gave the corresponding hydrazide, does not m. 330°. Hydrogenation of 4-methyl-2,6-dichloropyrimidine in aq. KOH over Raney Ni at 90 atm. gave 45% *4-methylpyrimidine* in the aq. soln. which oxidized with $KMnO_4$ to 54% *pyrimidine-4-carboxylic acid*, m. 238-40°. This treated with $SOCl_2$ followed by EtOH, gave 84.6% *Et ester*, b_{18} 155-60°, m. 37-9°, readily converted to the hydrazide, m. 147-7.5°. I refluxed briefly with MeOH-$(CH_2NH_2)_2$ gave *di(2-pyrazinoylamino)ethane*, m. 296-7° (from 50% AcOH). The hydrazides heated briefly with the given aldehydes gave the corresponding *hydrazones* (yield and m.p. shown): I *hydrazide*: *4,3-HO(MeO)C_6H_3-CHO* (II), 86.5%, 241-2°; *p-HOC_6H_4CHO* (III), 97%, 290-1°; and *2,3,4-$HO_3C(MeO)_2C_6H_4CHO$*, 75% 217-18°. *3-Pyridazone-6-carboxylic hydrazide*: III, 80%, infusible; *p-AcNHC_6H_4CHO*, 77.5%, 330°; II, 80.6%, 296-8°; *p-$Me_2NC_6H_4CHO$* (IV), 84%, 285°. *Pyrimidine-4-carboxylic acid hydrazide*: II, —, 252-3°; III, —, 285-6°; IV, —, 288-9°. I with $AcNHCH_2CH_2NH_2$ gave 38% $C_8H_{12}O_2N_4$, m. 187-8°, while *o*-$C_6H_4(NH_2)_2$ with 2-pyrazinecarbonyl chloride (V) gave $C_{11}H_{10}ON_4$, m. 199-200° (Ac deriv., m. 105-7°). V with *m*-$O_2NC_6H_4NH_2$ gave 79% $C_{11}H_8O_3N_4$, m. 176-7°, while *p*-$AcNHC_6H_4NH_2$ gave 83% $C_{13}H_{12}O_2N_4$, m. 229-30°. The amides and hydrazones listed above show no antibacterial action except for a slight activity against tuberculosis bacteria.

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Glyoxaline. II. 4(5)-Phenylglyoxalin-2-yl alkyl (aryl) sulphones and sulphoxides. P. M. Kochergin and M. N. Shchukina (*Zh. obshch. Khim.*, 1955, 25, 2318—2323).—A series of 4(5)-phenylglyoxalin-2-yl alkyl (aryl) sulphones and sulphoxides were obtained which showed varying degrees of antibacterial power. The formation of sulphoxides and sulphones depends on the amount of H_2O_2 used, and proceeds in stages. By the action of equimol. proportions of H_2O_2 on 4(5)-phenyl-2-*iso*pentyl- and 4(5)-phenyl-2-*n*-hexyl-thioglyoxaline at room temp., sulphoxides with yields of 98% were obtained; with 2 mol. H_2O_2 and 1 mol. of sulphide in similar conditions (15—45°) sulphones were obtained in 70—94% yields. 2-*iso*Pentylsulphinyl-4(5)-phenylglyoxaline hydrochloride exhibited anti-staphylococcal activity (1 : 4000) and the 2-*n*-hexylsulphinyl deriv. anti-anthraxoid activity (1 : 8000). Sulphones derived from glyoxalines showed less activity in osteo-tuberculosis treatment than did sulphides; sulphoxides showed the greatest activity.

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Structure of pyrazine derivatives formed by condensation of aminomalonic acid diamide with methylglyoxal. T. V. Gostinskaya and M. N. Shchukina. J. Gen. Chem. U.S.S.R. 25, 2425-7(1955)(English translation).—See C.A. 50, 9430d. B. M. R.

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Characteristics of pyrazine derivatives obtained by condensation of diamides of aminomalonic acid with methylglyoxal. T. V. Gortinskaya and M. N. Shchukina (*Zh. obshch. Khim.*, 1955, 25, 2529—2531).—It was found that by condensation of methylglyoxal with diamides of aminomalonic acid, amides of 2-hydroxy-6-methylpyrazine-3-carboxylic acid were formed in good yields and not 2-hydroxy-5-methylpyrazine-3-carboxylic acid as stated in earlier research work by Karmas and Spoerri (*J. Amer. chem. Soc.*, 1952, 74, 1580) and Jones (*ibid.*, 1949, 71, 78). A. L. B.

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Antituberculous chemiotherapeutic preparations. M. N. Shchukina and T. P. Sycheva. *Khim. Nauka i Prom.* 1, 419-32(1956).—Review with 109 references through 1955 covering advances in tuberculosis chemotherapy, including Au derivs., sulfa drugs, sulfones, *p*-aminosalicylic acid and its derivs., streptomycin and other antibiotics, thiosemicarbazones and derivs. of isonicotinic acid as well as hydrazides and amides of carboxylic acids and alkylthiohydantoins, along with numerous miscellaneous substances.
—C. M. Kosolapoff

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