"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, J.

POLAND / Pharmacology, Toxicology. General Problems U-1

Abs Jour : Ref. Zh.-Biol, No 2, 1958, No 7875

Author : Skovronskaya - Serafimova B., Venulet, J.

Inst :

Title : Chemical Structure and Biological Action.

Orig Pub : Wiadom. Chem., 1955, 9, No 12, 597-615

Abstract : The similarity of spatial configurations of stilbesterol and estrone probably explains the biological similarity of their actions. The chemical structure of a substance accounts for its physico-chemical characteristics which, to a great extent, influence its biological activity. The biological activity in a homologous series frequently changes with an increase in chain length and with an associated alteration in the physical pecularities of the homologues. By

Card : 1/2

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

USSR/Pharmacology. Toxicology. Chemotherapeutical V
Preparations

Abs Jour : Ref Zhur-Biol., No 8, 1958, 37706

Author : Venulet I., Yakimovskaya K., Urbanskaya A.
Inst : Not given
Title : Conduct of Hydroxamic Acids in Animal Organisms (Povedeniye gidroksamovych kislot v zhivotnykh organizmakh)

Orig Pub : Byul. Pol'skoy AN, 1956, Otd. 2, 4, No 5, 195-201

Abstract : The blood content of salicyl hydroxamic acid (1) 5-brom-salicyl hydroxamic acids (11) was determined colometrically. 1 and 11 were found in the blood in the amount of 16 mg/ml in the first 4 minutes after their administration in a dose of 0.05 g/km the color reaction disappeared 25-30 minutes later. In large quantities both preparations (50%) combine with erythrocytes, and in 30%

Card 1/2

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USSR/Pharmacology. Virology. Chemotherapeutical Preparations

Abs Jour : Ref Zhur-Biol., No 8, 1958, 37705

Abstract : quantities with the proteins. I and II, however, rapidly disappear from blood circulation. Their disappearance is apparently connected with the metabolism of the hydroxamic acids in the organism. In addition the authors established that the liver and to a lesser degree the renal organs actively decompose I and II.

Card 2/2

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, J.

Współczesne problemy farmkoterapii (Contemporary problems of pharmacotherapy), by J. Venulet. Reported in New Books, (Nowe Książki), No. 6, March 15, 1956.

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

GAWECKA, I.; SZMAL, Z.; VENULET, J.

Effect of physostigmine on action of pendiomide. Acta physiol. polon. 7 no.3:351-358 1956.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie Kierownik: dr. J. Venulet.
(PHYSOSTIGMINE, effects,
on reactivity to pendiomide (Pol))
(AUTONOMIC DRUGS, effects,
pendiomide, eff. of physostigmine on reactivity (Pol))

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, Jan

Recent concepts on action of bacterial pyrogens. Polski tygod. lek. 11 no.40:1719-1722 1 Oct 56.

1. Adres: Warszawa, ul Sandomierska.
(PYROGENS, effects,
concepts of mechanism of action of bact. pyrogens (Pol))

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, J.

POLAND/General Problems of Pathology - Pathophysiology of the Infectious Process. T-4

Abs Jour : Ref Zhur - Biol., No 1, 1958, 3037

Author : Venulet, J.
Inst : -

Title : A New Outlook on Bacterial Pyrogens.

Orig Pub : Farmac. Polska, 1956, 12, No 6, 144-146

Abstract : Administration of somatic antigen from a number of gramnegative bacteria caused an elevation in animals' temperature. Lipid A (phospholipid), and not the protein component of an antigen, had this pyrogenic effect. The phospholipid had a high biological activity. For example, coliform bacilli preparations in a dosage of 0.0012-0.003 gamma/kg caused a temperature elevation of 0.6° C. The rise in temperature began after 1-2 hours and persisted for 8-12 hours. It was caused by an insignificant rise in metabolic rate and, chiefly, by a decrease in heat loss due to cutaneous

Card 1/3

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

POLAND/General Problems of Pathology - Pathophysiology of the Infectious Process. T-4

Abs Jour : Ref Zhur - Biol., No 1, 1958, 3037

vasoconstriction. At first there was a leucopenia, which was followed by a leucocytosis. Intravenous administration of a blood-pyrogen mixture that had been previously incubated shortened the latent period and caused a rapid elevation of temperature. The phospholipid had probably combined with an euglobulin of the blood. Pyrogens have a low level of toxicity; their lethal dose for the rabbits was a million times that causing a pyrogenic effect. Under the pyrogen's influence the body's resistance increased and there was an improvement in various diseases. Their effects resembled those of cortisone and ACTH, but differed in many respects; e.g. unlike the hormones, the pyrogens favored healing. Pyrogens were successfully used to treat gastric ulcer, to promote regenerative processes in the central and peripheral nervous systems, and in cutaneous inflammations of a trophic or allergic character.

Card 2/3

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

POLAND/General Problems of Pathology - Pathophysiology of the Infectious Process. T-4

Abs Jour : Ref Zhur - Biol., No 1, 1958, 3037

In addition, although they are not proteins, pyrogens can be used in protein replacement therapy and should be used in lieu of panodin, Delbet vaccine and milk injections.

Card 3/3

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, J.

KIEDRZYNSKI, Z.; VENULET, J.

New method of measurement of heat loss. Acta physiol. polon. 8 no.3: 377-378 1957.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie. Kierownik: dr J. Venulet.

(BODY TEMPERATURE,
heat loss, measurement (Pol))

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, Jan; DESPERAK, Anna

Certain pharmacological properties of pyrogen of E. coli. Med. dosw. mikrob. 9 no.3:253-258 1957.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie. Kierownik: dr med. J. Venulet.

(ESCHERICHIA COLI,
pyrogens, pharmacol. (Pol))
(PYROGENS, preparation of,
from E. coli, pharmacol. (Pol))

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

1471. THE METABOLIC EFFECT OF A LIPOPOLYSACCHARIDE EXTRACTED FROM ESCH. COLI - Über die Stoffwechselwirkung eines Lipopolysaccharids aus Escherichia coli - Venulat J. and Desperak A. Anst. fur Pharmakol., Arzneimittelforsch. -Inst., Warschau - EXPERIENTIA (Basel) 1957, 13/9 (364-365) Tables 2

Rabbits were injected intravenously with 0.01 μg. per kg. of a lipopolysaccharide extracted from E. coli by the Westphal method. It has a strong pyrogenic activity. Injection caused hyperglycaemia and decrease of the concentration of lactic acid in the blood. The oxygen consumption of the livers of the treated animals increased considerably. This was also observed in vitro when the pyrogenic substance was brought into contact with renal and hepatic tissues. Cavallo - Naples

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, JAN

JAKIMOWSKA, Krystyna; VENULET, Jan

Effect of antituberculous agents & other preparations on egg-white edema. Gruzlica 25 no.12:959-967 Dec 57.

1. Z Instytutu Gruzlicy Dyrektor; prof. dr J. Misiewicz i z Zakladu Farmakologii Instytutu Lekow w Warszawie Kierownik Zakladu: dr med. J. Venulet. Adres: Warszawa, ul. Chelmska 34.

(EDEMA, exper.
induced by egg white, eff. of antituberc. agents & other drugs in rats (Pol))

(EGG WHITE, eff.
induction of edema, eff. of antituberc. agents & other drugs in rats (Pol))

(TUBERCULOSIS
antituberc. agents, eff. on edema induced by egg white in rats (Pol))

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

POLAND / Pharmacology and Toxicology. Chemotherapeutic Agents. V-10

Abs Jour : Ref. Zhur - Biologiya, No 17, 1958, No. 80709

Author : Urbanska, Alicja; Venulet, Jan
Inst : Not given
Title : Influence of Pharmacological Drugs on the Content of Isoniacid in the Blood and Tissues

Orig Pub : Gruzlica, 1957, 25, No 12, 969-976

Abstract : The influence was studied of histamine (I), antistin (II), largactyl (III), doril (IV) and pyrogen (V) on the content of isoniacid (IS) in the blood and tissues of the lungs, liver and kidneys. The greatest decrease of the content of IS in the blood set in under the influence of I; the weakest effect was from V, III and IV. Increase of the content of IS in the tissues occurred under the influence of U and IV; in lesser degree, from III and I. The influence of II did not change the content of IS in the blood and organs.

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

URBANSK, Tadeusz; BELZECKI, Czeslaw; CHECHELSKA, Bozena; CHYLINSKA, Barbara; DABROWSKA, Halina; FALECKI, Jerzy; GURNE, Daniela; HALSKI, Leszek; MALINOWSKI, Stanislaw; SERAFINOWA, Barbara; ZYLOWSKI, Jerzy; SLOPEK, Stefan; KAMIENSKA, Irena; VENULET, Jan; JANOWIEC, Mieczyslaw; JAKIMOWSKA, Krystyna; URBANSKA, Alicja; KOZNIEWICOW, Anatol

Searching for new anti-tuberculosis drugs. Gruzlica 26 no.11:889-917 Nov 58.

1. Z Zakladu Syntezy Lekow Instytutu Gruzlicy Kierownik Zakladu: prof. dr T. Urbanski Dyrektor Instytutu: prof. dr J. Misiewicz Pracownia Synt. Lekow Przeciwgruzlicych, Warszawa, ul. Koszykowa 75.

(TUBERCULOSIS, therapy,
investigation of 300 cpds. for anti-tuberc. eff. (Pol))

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

JANOWIEC, Mieczyslaw; VENULET, Jan

Experimental ocular tuberculosis in rabbits. Gruzlica 27 no.3:213-216 Mar 59.

1. Z Zakladu Farmakologii Instytut Lekow w Warszawie Kierownik: dr med. J. Venulet. Adres: Warszawa, ul. Chelmska 30/34.
(TUBERCULOSIS, OCULAR, experimental, in rabbits (Pol))

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

JAKIMOWSKA, Krystyna; WITKIEWICZ, Maria; VENULET, Jan

Pharmacology of p-nitrophenylguanylurea (T-72). Acta physiol. Pol. 15 no.5:701-712 S-O '64

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie (Kierownik: doc. dr. J. Venulet).

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

JANOWIEC, Mieczyslaw; VENULET, Jan

Experimental tuberculosis in hooded rats. Med. dosw. mikrobiol. 16 no.1:13-20 '64.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie (Kierownik: doc. dr. J. Venulet).

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

JANOWIEC, Mieczyslaw; WOJCIK, Ryszard,A.; VENULET, Jan

Associated effect of isonicotinic acid hydrazide (INH) and of some drugs influencing the macro-organism in experimental tuberculosis. Med. dosw. mikrobiol. 15 no.4:311-315 '63.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie (kierownik:doc.dr.med. J. Venulet) i z Katedry Statystyki Matematycznej SGGW w Warszawie (kierownik: prof.dr. Z. Nawrocki).

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

JANOWIEC, Mieczyslaw; VENULET, Jan

Antitubercular effects of ethyl acetoacetate isonicotinoylhydrazone (T-428) administered jointly with ultracortenol and somatotropin. Med. dosw. mikrobiol. 15 no.4:317-319 '63.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie;kierownik: doc.dr. J.Venulet.

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

JANOWIEC, Mieczyslaw; VENULET, Jan

Experimental tuberculosis in rats. Gruzlica 31 no.6:712-716 Je'63.

1. Zaklad Farmakologii Instytutu Lekow, Warszawa.

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

LANGE, Jerzy; URBANSKI, Tadeusz; VEHULET, Jan

Preparation and biological activity of the derivatives of phenylsuccinic acid. Pts.2-3. Rocz. chemii 36 no.11:1625-1638 '62.

1. Department of Organic Technolgy II, Institute of Technology, Warsaw.

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

LANGE, Jerzy; URBANSKI, Tadeusz; VEMULET, Jan

Preparation and biological activity of the derivatives of phenylsuccinic acid. III. Rocz chemii 36 no.11:1631-1638 '62.

1. Department of Organic Technology 11, Institute of Technology, Warsaw.

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

DESPERAK-NACIAZEK, Anna; JAKIMOWSKA, Krystyna; JANOWIEC, Mieczyslaw; KRYSICKA-DOCZKAL, Halina; PAWLIKOWSKI, Leszek; VENULET, Jan

Chemotherapeutic and pharmacological properties of the sodium salts of m-aminosalicylic acid. Med. dosw. mikrobiol. 15 no.1:85-90 '63.

1. Z Zakladu Farmakologii Instytutu w Warszawie Kierownik: doc. dr J. Venulet.

(AMINOSALICYLIC ACID) (PHARMACOLOGY)
(MYCOBACTERIUM TUBERCULOSIS) (TUBERCULOSIS)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, J.

3

POLAND

DESPZAM-NACIAZEK, Anna, JAZIMOWSKA, Krystyna, JENONIEC, Mieczyslaw, KRYSICKA-DOCZKAL, Halina, PAWLIKOWSKI Leszek, and VENULET, Jan, Department of Pharmacology (Zaklad Farmakologii), Drug Institute (Instytut Leków) in Warsaw (Director, Docent, Dr. J. VENULET)

"Chemotherapeutic and Pharmacologic Properties of Sodium Salts of M-Aminosalicylic Acid."

Warsaw, Medycyna Doswiadczalna i Mikrobiologia, Vol 15, No 1, 63, pp 85-90.

Abstract: [Authors' English summary] Investigations in vitro and in vivo revealed merely a weak action of the sodium salts of 5-aminosalicylic acid (MAS) upon tubercle bacilli, as well as on experimental tuberculosis in laboratory animals. When tested in vitro, MAS was similar in action but weaker than PAS. Both derivatives are less effective than isonicotinic acid hydrazide or Streptomycin. The toxicity of MAS is slightly higher than that of PAS preparations. All 6 references are by Polish authors (in 1 German, 1 French, and the rest in Polish publications or unpublished).

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APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

SZMAL, Zdislaw; VENULET, Jan

Effect of some quarternary ammonium compounds with sulfur in the aliphatic chain on myeoneural transmission. Acta physiol. polon. 13 no.6:717-728 '62.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie Kierownik: doc. dr J. Venulet.
(AMMONIUM COMPOUNDS) (SULFUR) (SYNAPSES)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

JANOWIEC, Mieczyslaw; VENULET, Jan

Effect of hydrazides of aspartic and glutamic acids on experimental tuberculosis. Med. dosw. mikrob. 14 no.3:247-252 '62.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie.
(ASPARTIC ACID rel cpds) (GLUTAMATES pharmacol)
(TUBERCULOSIS exper)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, Jan, z pomoca techniczna; KALINOWSKIEJ, Marii

On the correlation between anti-serctonin and depressive effects of certain phenothiazine derivatives. Acta physiol pol 12 no.2: 281-290 '61.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie Kierownik: doc. dr J. Venulet.
(REFLEX CONDITIONED pharmacol) (SEROTONIN antag)
(CHLORPROMAZINE pharmacol) (TRANQUILIZING AGENTS pharmacol)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, Jan

Psychopharmacology. Acta physiol. polon. 12 no.6:915-927 '61.
(PSYCHOPHARMACOLOGY)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

JANOWIEC, Mieczyslaw; VENULET, Jan

Antituberculous properties of ethyl acetoacetate isonicotinoylhydrazone (preparation T-428). II.

1. Z Zakladu Farmakologii Instytutu Lekow w Warszawie Kierownik: doc. dr med. J. Venulet.

(ISONIAZID rel cpds)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENULET, Jan

Psychopharmacology. Acta physiol pol 12 no.6:915-927 '61.

1. Adres autora: Zaklad Farmakologii Instytutu Lekow, Warszawa 36, ul. Chelmska 30.

(PHARMACOLOGY) (PSYCHOLOGY, PATHOLOGICAL)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

SURNAME (in caps); Given Names

Country: Poland

Academic Degrees: Dr

Affiliation: [not given]

Source: Warsaw, Farmacja Polska, Vol XVII, No 15-16, 25 August 1961, pp 310-312

Data: "Pharmacological Investigations as an Approach to Clinical Tests."

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENUS, B. G.

Mineralogical correlation of loose Mesozoic and Cenozoic sediments in the Amur-Zeya Depression. Vest LGU 19 no. 6:71-82 '64. (MIRA 17:5)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENUS, B.C.; TYRIN, A.N.

Titanium potential of the present beaches of Lake Omega. Vest.AN (MIRA 18:8)
20 no.12:133-134 '65.

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

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CA

Transformation of asymmetric dicyclohexylethylene oxide. E. D. VENUS-DANILOVA. *J. Russ. Phys.-Chem. Soc.* 61, 1479–84(1929).—It was shown in the case of the asym. methylphenylethylene oxide that the doubly substituted asym. ethylene oxides can be isomerized either to an aldehyde or to a ketone depending on the working conditions (S. Danilov and E. D. Venus-Danilova, *Ibid* 59, 187(1927); *C. A.* 21, 2465). Asym. dicyclohexylethylene oxide (I) undergoes an analogous conversion to dicyclohexylacetaldehyde (II) which on treating with H_2SO_4 is isomerized to dicyclohexylethanone (III). $(C_6H_{11})_2C.CH_2.O \longrightarrow (C_6H_{11})_2CHCHO \longrightarrow C_6H_{11}CH_2COC_6H_{11}$.

Expt. part. *Dicyclohexyl ketone* (IV), obtained in 97% yield by oxidizing dicyclohexylcarbinol, m 61°, with $K_2Cr_2O_7$ in H_2SO_4 first at room temp. and then at 75°, b_{12} 148–8.5°. *Methyldicyclohexylcarbinol* (V), obtained in 92.5% yield by treating IV with Mg-MeI in Et_2O, b_{12} 156°. *Asym. dicyclohexylethylene* (VI), obtained in 21 g yield when 28 g. of V with 110 g. of $C_2O_4H_2$ in 80 cc. H_2O is simmered for 4 hrs in an oil bath, the product is steam distd., extd. with Et_2O, dried with calcined Na_2SO_4 and redistd. *in vacuo*, b_{12} 140°. *Asym. dicyclohexylethylene oxide*, obtained in 76% yield when 19 g. of VI in 30 cc. of $CHCl_3$ is treated for 7 days with 615 cc. of a soln. of H_2O_2H (contg. 2.3 g. of active O) in $CHCl_3$, the product is washed with NaOH, dried with K_2CO_3 and redistd., b_{12} 152–2.5°. *Isomerization of the oxide.* I is converted to II or III depending on the amt. and concn. of H_2SO_4 used in the reaction. II is obtained in 62% yield when 4 g. of I are refluxed for 50 min. with a small piece of pumice stone wetted with 60% H_2SO_4; after cooling the mass is dild. with Et_2O, washed with Na_2CO_3, dried and redistd., b_{12} 152–4.5°. III is prepd. in 0.8 g. yield, by dropping in the course of 70 min. 4 g. of I into 34 g. of H_2SO_4 (d. 1.84), cooled to −10°, the whole being stirred with a current of CO_2, and then allowed to stand for 3 hrs. at −8°, then the red liquor is poured on ice, extd. with Et_2O, washed with Na_2CO_3, dried with Na_2SO_4, the Et_2O expelled, redistd. *in vacuo*, the distillate b_{12} 150–62°. CHAS. BLANC

ASTIA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

BC

Isomerization of hydroxyaldehydes. II. Transformation of bromodicyclohexylacetaldehyde and dicyclohexylglycolaldehyde. S. DANILOV and E. VENUS-DANILOVA (J. Russ. Phys. Chem. Soc., 1929, 61, 1661—1662).—See A., 1929, 1448.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

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CA

Isomerization of the hydroxy aldehydes. III. Transformation of glucose into ketose (fructose). S. DANILOV, E. VENUS-DANILOVA AND P. SHANTAROVICH. *J. Russ. Phys.-Chem. Soc.* 62, 494(1930); *Ber.* 63B, 2269-74(1930); cf. *C. A.* 24, 1093.—Glucose heated in org. bases, especially pyridine and quinoline, gives neutral products consisting of only glucose and fructose without any admixed mannose. In aq. pyridine and aq. alc. quinoline the rearrangement does not proceed so smoothly, 1-2% of org. acids is formed and mannose could be detected. C. A. R.

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Isomerization of the hydroxy aldehydes. IV. Transformation of benzylbromoacetaldehyde and benzylglycolaldehyde. S. Danilov and E. Venus-Danilova. *Ber.* 63B, 2765-75; *J. Russ. Phys.-Chem. Soc.* 62, 1697-1711(1930); cf. *C. A.* 24, 1003.—*Benzylbromoacetaldehyde* (I), from $PhCH_2CH_2CHO$ and Br in CS_2, is an unstable oil readily resinifying and polymerizing on standing, but it yields a cryst. *monohydrate*, m. 87.5-8°, which permitted of studying the conversion of I into *benzylglycolaldehyde* (II) and the acid (III). Heated with H_2O and freshly ppted $BaCO_3$ I gives II in quite good yields, together with 4% $PhCH_2CH_2CO_2H$ (IV). With Ag_2O are obtained 20% II, 9.4% III, 5.6% of a condensation product (V) of I and 31% IV. PbO_2 yields chiefly halogen contg. condensation products of I and 22.6% IV. The structure of II is established by its prepn., its deriv. and its oxidation products, $KMnO_4$ in aq. C_6H_6 gives $PhCH_2CHO$, $BzOH$ and III. Its isomerization into the HO ketone is accompanied by the formation of a diketone: $PhCH_2CH(OH)CHO$ (II) ⟶ $PhCH(OH)COMe$ (VI) + $PhCOCOMe$ (VII). II, b_{14} 120-1°, m. 51.5-2°, reacts with NH_3-$AgNO_3$, fuchsin-SO_2 and Fehling soln., mol. wt. in freezing C_6H_6 143-7, *oxime*, m. 123°; *semicarbazone*, m. 136.5°, *phenylosazone*, yellow, m. 137°, *benzoate*, m. 70°, mol. wt. in freezing C_6H_6 252-6. III, m. 97.5°, mol. wt. in freezing C_6H_6 130. VII, b_{14} 113.5°, dioxime, m. 238-9°. VI, b_{14} 130-2°, mol. wt. in freezing C_6H_6 155-61, semicarbazone, m. 194°. With $PhMgBr$ VI yields $PhMeC(OH)CH(OH)Ph$, m. 103°, gives with concd. H_2SO_4 a deep red color destroyed by H_2O and is oxidized by CrO_3-AcOH to $PhCOMe$ and $BzOH$. C. A. R.

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

1A

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Hydration of acetylene alcohols with a primary alcohol group. E. D. VENUS-DANILOVA AND S. N. DANILOV. *J. Gen. Chem.* (U. S. S. R.) 2, 645-58(1932).—The synthesis of the primary α-keto alcs. was undertaken to clarify the interrelation of the isomeric phenylacetol, $PhCH_2COCH_2OH$ (I) and phenylglycolaldehyde, $PhCH_2CH(OH)CHO$ (*C. A.* 25, 1819). All syntheses were carried out by hydration in the presence of $(AcO)_2Hg$. It was assumed that the addn. of H_2O to $PhC:CCH_2OH$ (II) and its homologs would result in formation of α- and β-keto alcs., but no α-keto alcs. were formed. With II was obtained mainly $BzCH:CH_2$ (III) and a little of $BzCH_2CH_2OH$ (IV), and with $PhC:CCH_2OAc$ a mixt. of $BzCH_2CH_2OAc$, III and its polymer. The formation of III is evidently caused by splitting off of H_2O from the intermediate IV. $Me_2CC:CCH_2OH$ (V) reacted analogously, producing $Me_2CCOCH:CH_2$ (VI) and $Me_2CCOCH_2CH_2OH$ (VII), and $Me_2CC:CCH_2OAc$ the corresponding acetates. The vinyl ketones and β-keto alcs. and their acetates in the mixts. were thus identified. The semicarbazone of IV, m. 170°, was obtained together with the semicarbazide semicarbazone of III, m. 206°; semicarbazone of VII m. 223° (decompn.), and that of the acetate m. 127°, while VI gave no semicarbazone. III produced 1,3-diphenylpyrazoline, m. 153.5-4°, and so did IV and its acetate. VII and VI (and its acetate) with $PhNHNH_2$ gave $Me_2CC(:NNHPh)CH_2CH_2NHNHPh$, m. 134°. III, with NH_2OH (1 mol. to 1 mol.) gave $C_{10}H_{12}O_2N$ (VIII), m. 122.5°, which, heated with 25% H_2SO_4 or 30% HCl, does not split the NH_2OH radical, but gives salts with 1 mol. of HCl or H_2SO_4. III, treated with an excess of NH_2OH, or VIII gently heated with NH_2OH in aq. alc., is converted to $C_{10}H_{12}O_2N_2$ (IX), m. 153°, which, treated with mineral acids, gives no salts but splits off 2 NH_2OH radicals with formation of VIII. The proposed structural formulas are for VIII $(PhCOCHMe)_2NOH$ and for IX $[PhC(:NOH)CHMe]_2NOH$.

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PROCESSES AND PROPERTIES INDEX

CA

Isomerization of hydroxy aldehydes. V. Oxidative-reductive transformations of α-hydroxyisobutyraldehyde. B. Danilov and K. Venus-Danilova. *Ber.* 67B, 24-35 (1934); *J. Gen. Chem.* (U. S. S. R.) 3, 559-72(1933); cf. *C. A.* 25, 1819.—Mono- or polymeric $Me_2C(OH)CHO$ (I) in alk. soln. (180 hrs. in 3% NaOH at 20°, long standing at room temp. or 3 hrs. at 100° in 3-4.4% KOH) gives only a mixt. of $Me_2C(OH)CO_2H$ and $Me_2C(OH)CH_2OH$ with or without unchanged I, i. e., it undergoes almost solely the Cannizzaro reaction. In the presence of $Cu(OH)_2$ or $Pb(OH)_2$ at 100°, however, there is obtained, in addn. to unchanged I and the rearrangement product, $MeCH(OH)COMe$ (II), 40-5% Me_2CHCO_2H (with the Cu salts a little (7-8%) $Me_2C(OH)CO_2H$ is also formed). The metal oxide transports the O from the OH to the CHO group but is itself not reduced. The oxidative-reductive transformation of I into II is best effected (with almost quant. yield) by heating 6-8 hrs. with very dil. acid at 185°. In alk. soln. II is formed only in very small amt. and in the presence of oxides of heavy metals. No Ac_2 could be detected, showing that HO ketones with alkyl radicals are oxidized to diketones with considerably greater difficulty than those which contain Ph or cyclohexyl residues. I was prepd. from polymeric Me_2CBr-CHO made directly from Br and polymerized Me_2CH-CHO

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CHO. After several careful distns. it bs 62-4° and on standing gradually thickened, finally solidifying after some months to crystals m. 78-4°, the mol. wt. in freezing C_6H_6 increasing from 95.1 immediately after distn. to 226 in 48 hrs., when I showed d_4^{20} 1.1014, d_4^{40} 1.0930, n_D^{20} 1.44181, n_D 1.43070; semicarbazone, m. 164-5°; p-nitrophenylhydrazone, m. 153-9° (decompn.). C. A. R.

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

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Syntheses of acroleins with cyclohexyl and aromati radicals. K. D. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 4, 866-70(1934).—Cyclohexylacrolein, C_6H_{11}·CH:CHCHO (I), b_4 92-4°, is obtained when 10 g. hexahydrobenzaldehyde (Wood and Comley, *C. A.* 18, 668), b_{10} 63-4°, in 6 parts of MeOH and 5 parts of H_2O at 70° is treated in a N atm., with shaking, during 5 hrs. with 25% excess of 6% AcH in 5-cc. portions and 5% NaOH in 0.5-cc. portion to maintain a slightly alk. reaction. The residue from the Et_2O extn. is fractionated at 50-150°. I is easily polymerized to a glass-like yellow mass. I and its polymer have an offensive odor. Semicarbazone of I, m. 179-80°. I is oxidized with $KMnO_4$ to C_6H_{11}-CO_2H, m. 30-1°, and with Ag_2O to $C_6H_{11}CH:CHCO_2H$, m. 58-9°. *Piperonylacrolein*, $CH_2O_2C_6H_3CH:CHCHO$, m. 86-6.5° (alc.), is obtained in 16% yield when to piperonal, m. 37°, in 4 parts of MeOH are added with stirring 40 parts of H_2O and then, in small portions, 20% AcH and 10% NaOH as described above; after continued stirring for 2-3 hrs., the mixt. is acidified with AcOH, extd. with Et_2O, the Et_2O and MeOH are evapd. at 30-5°, and allowed to stand for 3-4 days for crystn. Semicarbazone, m. 234° (alc.) Chas. Blanc

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E. D. VENUS-DANILOVA

Aldehydes and hydroxy aldehydes of the polymethylene series. I. Conversion of hexahydrobenzaldehyde. E. D. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 6, 691-700 (1936); *J. C. A.* 30, 3152[illegible]. Previously it was shown that open-chain aldehydes can be catalytically isomerized into ketones at elevated temps. (Danilov and V.-D., *C. A.* 20, 1988; 21, 2120; 22, 950). In the case of cyclic aldehydes it could be expected that the ketonic conversion would be accompanied by cyclic isomerization. Hexahydrobenzaldehyde (I) could be converted into suberone or acetylcyclopentane (II). In all cases of isomerization only II and no suberone was formed. The best results with 57% II were obtained by heating 3 g. I, 1.8 g. H_2SO_4, 18 g. H_2SO_4 (d. 1.84), 15 cc. of 90% alc. and 2 cc. H_2O in a sealed tube (filled with CO_2) at 142-4° for 5 hrs. II was isolated as the semicarbazone from the Et_2O ext. of the steam-distn. residue. Treating I with concd. H_2SO_4 at −10° and with 40% H_2SO_4 + alc. by heating, without $HgSO_4$, gave 40-3% II, contaminated with condensation and polymerization products of I. Heating I in dil. alc. in the presence of $HgCl_2$ or $HgBr_2$ gave 4-13% II with considerable I unchanged. Substituting C_5H_5N for alc. resulted in 32% II. The united fractions of the semicarbazone of II, m. 144-5°, decompd. by boiling with 20% $(CO_2H)_2$, gave 100% II, b. 158-9.5°, b_{50} 75-6.5°, d_4^{20} 0.9269, d_{20}^{20} 0.918, n_D^{20} 1.444. II oxidized with alk. Br in H_2O gave cyclopentanecarboxylic acid. The amide m. 178-9°.

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Aldehydes and hydroxy aldehydes of the polymethylene series. II. Products of cyclopentanone condensation

E. D. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 6, 917-21(1936); cf. *C. A.* 30, 6341[9].—Attempts to reduce cyclopentanone (I) with $ClMgCMe_3$ to cyclopentanol (II) (cf. Danilov, *J. Russ. Phys.-Chem. Soc.* 59, 1110(1927)) resulted in the formation of 11.6% II and 48% cyclopentylidene-2-cyclopentanone (III) (cf. Meerwein, *C. A.* 8, 3183). In the reduction of I with Na in Et_2O (Wislicenus, *Ann.* 275, 322(1893)), a part of I is condensed to cyclopentyl-2-cyclopentanol (IV), a product of the reduction of the intermediate III. Harries and Wagner (*C. A.* 9, 2877) assumed that this condensation results by the action of EtONa formed with the contaminating EtOH in the Et_2O. In fact, by repeating the expts. with Et_2O free from EtOH, the yield of II was increased to 44%. A similar improvement was effected by removing twice the lower satd. layer of NaOH and adding an equal part of H_2O and fresh Et_2O. This method gave 48-50% II, 46% IV, b_{14} 119-20°, d_4^{20} 0.9945, d_4^{40} 0.985, and dihydroxybicyclopentyl (pinacol), m. 109-10°. III, b_{14} 123-3° (semicarbazone, m. 124°; oxime, m. 123°), reduced in dil. alc. with H_2 in the presence of Pt black gave 91% cyclopentyl-2-cyclopentanone (V), b_{17} 120-1°, b. 230-2°; d_4^{20} 0.9901, d_4^{40} 0.985; semicarbazone, m. 200°; oxime, m. 78°. IV (6 g.) with 20 g. Ac_2O and 8 g. of anhyd. NaOAc boiled for 2 hrs. gave 80% of the acetate, b_{14} 121-2°, d_4^{20} 0.9973, d_4^{40} 0.9882. IV oxidized with $K_2Cr_2O_7$ and H_2SO_4 in H_2O (Harries and Wagner, *loc. cit.*) gave 88% V. About 30 references.

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1ST AND 2ND CODES

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3RD AND 4TH CODES

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Aldehydes and hydroxy aldehydes of the polymethylene series. III. Conversion of cyclopentylformaldehyde. K. D. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 6, 1757-65(1936); cf. *C. A.* 31, 6654. Cyclopentylformaldehyde (I) in concd. H_2SO_4 undergoes isomerization to cyclohexanone (II) and its condensation products, 1-cyclohexylidene-2-cyclohexanone (III), dicyclohexylidenecyclohexanone (structure not definitely established) (IV), and dodecahydrotriphenylene (V). Ketones with 4-membered rings were not found in the reaction products, which agrees with the greater stability of 5- and 6-membered nuclei. I, b_{16} 52-3° (polymer, needles, m. 117-17.5°) is prepd. in 3.4% yield from cyclopentanone (VI) by the method of Darzens and Lefebure (*Compt. rend.* 142, 714(1906)) or from VI through the acetal in 13.5% yield by Wood and Comley's modification (*C. A.* 18, 658[8]) of the Chichibabin method. Treated with concd. H_2SO_4 at −16° with vigorous stirring I gives 0-8% II, b_{16} 62-4° (oxime, m. 89-90.5°; semicarbazone, m. 167.5-8.0°); 29-34% III, b_{16} 134-6° (oxime, m. 145-8°; semicarbazone, m. 176-7°); 27-40% IV, yellow sirup-like liquid, b_{16} 204-7°; and 0-3% V, m. 230-5°. Freshly distd. II under the same conditions gives 3.3-19% unchanged II, 34-9% III, 24-30% IV, and 0-1.4% V. Twenty-five references. IV. **Isomeric conversion of α-hydroxycyclopentylformaldehyde.** *Ibid.* 1784-95.—The isomeric transformation of α-HO aldehydes of the polymethylene series has not been studied. Study of α-*hydroxycyclopentylformaldehyde* (I), as the 1st member of the series, is of interest from the viewpoint of isomerization of cyclic hydroxycarbonyl compds. and from the viewpoint of studying the stability of cyclic systems with 4-, 5- and 6-membered rings. On the basis of exptl. data it is established that I undergoes transformation in either alk. or acid medium to form the keto alc. adipoin (1-cyclohexanol-2-one) (II) and cyclopentylformic acid (cyclopentanecarboxylic acid) (III). The formation of a keto alc. with a 4-membered nucleus could not be shown. Cyclopentylformaldehyde, prepd. through the acetal as described before, treated in CS_2 at 0° with 1 mol. Br gives 79.5% crude *α-bromocyclopentylformaldehyde (polymer)*, needles, m. 212-5° (decompn.)), which, suspend. with $BaCO_3$ on the boiling water bath for 40.5 hrs., gives 30% I, b_{16} 94-9°; the latter polymerizes very readily to a cryst. dimer, m. 95-7°, which partially depolymerizes when heated in AcOH or C_6H_6. The structure of I is proven by oxidation with moist Ag_2O to α-hydroxycyclopentanecarboxylic acid (IV), m. 103°. Neither the liquid form of I nor its dimer give a cryst. oxime or phenylhydrazone; treated with semicarbazide soln., both forms give a substituted *as-triazine*, probably *5-cyclopentyl-1,3,6-triazine*, white powder, difficultly sol. in alc., m. 216-18° (decompn.). Either I or its dimer heated in alc. with 1% H_2SO_4 for 5 hrs. at 135-8° in a sealed tube gives a mixt. of 80-7% II (keto form, b_{16} 78-81°; enol form, m. 123-6°; phenylhydrazone, m. 120-8°; phenylosazone, golden yellow needles, m. 152-3°); II with alk. $KMnO_4$ gives adipic acid, m. 148-8.5°.

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Heated for 3 hrs. on the boiling water bath with 2.5 10% KOH in the presence of $Pb(OH)_2$ or $Cu(OH)_2$ I or its polymer gives 15–24% II, 30–37% III, b_{10} 102° (amide, m. 178–9°), 0–22.5% 1-methylol-1-cyclopentanol, m. 39–40°; and 10–31% IV. III and IV are the products of a Cannizzaro reaction on I in the presence of alkali. IV is also formed by oxidation of I in the air or by $Cu(OH)_2$. Twenty seven references. V. **Bromo- and hydroxy-hexahydrobenzaldehyde.** *Ibid.* 1863-9.—Hexahydrobenzaldehyde in 9–10 vols. CS_2, treated at 0° with 1 mol. Br_2 in 4–5 vols. CS_2, gives 74% *α-bromohexahydrobenzaldehyde* (I), clear yellow oil, b_{10} 87–93°, which on standing 4 days polymerizes completely to the *trimer*, crystals from $CHCl_3$, m. 146–7° (partial decompn.). I with moist Ag_2O in alc.-water medium, heated 8 hrs. at 115–20°, gives 70% cyclohexanecarboxylic acid (II), m. 30°; treated with excess $BaCO_3$ on the boiling water bath for 50–60 hrs. I gives *α-hydroxyhexahydrobenzaldehyde* (III), b_{10} 102–8°, with some Δ^1-tetrahydrobenzaldehyde (IV), b_{10} 61–3° (semicarbazone, m. 212°), and II. III begins to polymerize immediately to the *dimer*, leaflets from C_6H_6, m. 126–7°, which in pyridine with $KMnO_4$ gives 71% α-hydroxyhexahydrobenzoic acid, m. 106–6.5°. Freshly distd. III with semicarbazide soln. in the cold gives the semicarbazone, m. 159–60°; heated in a sealed tube with alc.-aq. semicarbazide soln. for 2 hrs. at 110°, III or its polymer gives a light yellow powder, m. 221–3° (decompn.), supposedly *5-cyclohexyl-1,2,4-triazine*. IV with 1 aq. p-$O_2NC_6H_4NHNH_2.HCl$ gives the pyrazoline, C_6H_{10}-$CH:N.NC_6H_4NO_2$ red powder, m. 184° (decompn.).

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1ST AND 2ND ORDERS

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3RD AND 4TH ORDERS

Aldehydes and hydroxy aldehydes of the polymethylene series. VI. Isomeric changes of α hydroxyhexahydrobenzaldehyde. R. D. Venus Danilova and V. F. Kazimirova. *J. Gen. Chem.* (U. S. S. R.) 7, 2639-48 (in French 2648) (1937); cf. C. A. 31, 4280[8], 4281[8]. When this aldehyde is heated in an acid medium it isomerizes to α-hydroxysuberone (I). If MeOH or EtOH are present, α-methoxy- or α-ethoxysuberone are formed. In alk medium the yield of suberone deriv. is decreased and cyclohexane-carboxylic acid is also formed. 1-Methylol-1-cyclohexanol and α-hydroxycyclohexanecarboxylic acid are also formed by the Canizzaro reaction. In the presence of $Pb(OH)_2$ or $Cu(OH)_2$, the yields of these acids are increased. For comparison, I was prepd. from α-chlorosuberone and KOH. It b_{10} 130-2°, m. 27-9°, d_4^{20} 1.0361, n 1.46340, M. R. 33.86, parachor 298. With EtOH and HCl I gives *α-ethoxysuberone* b_{10} 75°, d_4^{20} 0.9337, n 1.43467, M. R. 43.55, parachor 394. H. M. Leicester

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1ST AND 2ND ORDERS

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The synthesis and properties of phenylcyclohexylacetaldehyde. F. D. Venus-Danilova and A. I. Bol'shakin. *J. Gen. Chem.* (U. S. S. R.) 7, 282-30 (in French 2830-1937). $PhCH_2CN$ is heated with $NaNH_2$ in C_6H_6 and treated with cyclohexyl bromide to give 36% of *phenylcyclohexylacetonitrile* (I), m. 55-6°. I does not react with $SnCl_2$ and dry HCl to give the aldehyde. However, when it is stirred with 85% H_2SO_4, it gives 32% of *phenylcyclohexylacetamide* m. 172-3°, and 57% of *phenylcyclohexylacetic acid* (II), m. 150-1°. II is also obtained in 91% yield when I is heated with HOAc and HCl at 180-200° for 24 hrs. in a sealed tube. With $CaCl_2$, II gives a monohydrated Ca salt. With $SOCl_2$ or PCl_5, II gives 76% of the *acid chloride* (III), b_{10} 157-8°, b_{15} 168-70°, d_4^{20} 1.1182, d_{20}^{20} 1.1049, d_4^{20} 1.1028, n_D^{20} 1.53604, M. R. 66.90, parachor 533.8. Shaking III with a limited amt. of H_2O gives the *acid anhydride*, m. 123-4°, which with KOH gives II. Reduction of III by H_2 in C_6H_6, using a Pd catalyst, gives *phenylcyclohexylacetaldehyde*, b_{10} 167-70°, d_4^{20} 1.0114, d_{20}^{20} 1.0057, d_4^{20} 1.001, n_D^{20} 1.53364, M. R. 62.80, parachor 496.8 (*semicarbazone*, m. 161-2°; *oxime*, m. 118°; *phenylhydrazone*, m. 122-3°). A by-product from the reduction of III is *methylphenylcyclohexylmethane*, b_{10} 132-3°, d_4^{20} 0.9624, d_{20}^{20} 0.9517, d_4^{20} 0.9503, n_D^{20} 1.53406, M. R. 61.56, parachor 481.0.

H. M. Leicester

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Aldehydes and hydroxyaldehydes of the polymethylene series. VII. By-products in the synthesis of ethyl tetramethylenedicarboxylate by the Kizhner method. E. D. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 8, 477-83 (in French 483)(1938); cf. C. A. 32, 2900[1]. Kizhner (*J. Russ. Phys.-Chem. Soc.* 37, 507(1905)) obtained 54% *di-Et cyclobutane-1,1-dicarboxylate* (I) from sodiomalonic ester (II) and $(CH_2)_3BrCl$ (III) with EtONa. The expts. were repeated by heating II (from 130 ml. of abs. alc., 11.5 g. Na and 58 g. of freshly prepd. Et malonate) with various amts. of III (up to 80 g.) at 70° for 1.75 hrs. and at boiling temp. for 2 hrs. After the addn. of EtONa (from 130 ml. alc. and 11.5 g. Na) and standing for 24 hrs., the reaction mixt. was refluxed for 5 hrs. and, after 24 hrs., the alc. was driven off at 50-60° and 35 mm. The residue was dissolved in water and extd. with Et_2O (ext. 1). The aq. layer on the addn. of excess H_2SO_4 was also extd. with Et_2O (ext. 2). The ext. 1 when freed from the Et_2O and steam distd. gave 8-10% I. The oily substance in the distn. flask was extd. with Et_2O and redistd. at 13 mm. and 195-205°, affording 23% *di-Et bis(γ-chloropropyl)malonate* (IV), m. 52-3° (alc.). IV with alk. KOH formed the HO compd.; this on heating at 140° and 18 mm. gave the *dilactone*, m. 49-51°. The latter gave IIa and Ag monolactone deriv. IV with EtONa gave identical results. The ext. 2 when distd. at atm. pressure is partially decompd., forming AcOEt. The distn. residue treated with KOH and steam distd. gave AcOH and the HO deriv. of IV. Chas. Blanc

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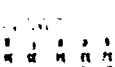
Aldehydes and hydroxyaldehydes of the polymethylene series. VIII. Isomeric changes of cyclobutylformaldehyde. E. D. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 8, 1179-91(1938); cf. *C. A.* 32, 7879[6].—$CH_2.CH_2.CH_2.CHCHO$ (I), b. 116-18° (*semicarbazone*, m. 119-20°), was obtained in 8-12.5% yield by (1) distg. equivs. of the Ca salts of cyclobutanecarboxylic and formic acids (cf. Colman and Perkin, *J. Chem. Soc.* 51, 238 (1887)), (2) oxidizing cyclobutylcarbinol with CrO_3 in the presence of $KHSO_4$ and (3) reducing cyclobutanecarbonyl chloride in C_6H_6 with H in the presence of Pd on $BaSO_4$ (cf. Rosenmund, *et al.*, *C. A.* 12, 2569). I is completely isomerized by heating it at 130-5° for 30 min. in the presence of pumice satd. with 60% H_2SO_4 and by refluxing it with $HgCl_2$ in 40 ml. of 50% alc. for 5 hrs. The reaction products are 30-55% *cyclopentanone* (II) and

45-70% $CH_2.CH_2.CH_2.CH.CH$ $CHCH.CH_2.CH_2.CH$



(cf. Shuikina, *C. A.* 31, 5332[6]). II, b. 127-33° (*semicarbazone*, m. 203°; *oxime*, m. 56°), reacts with BzH and concd. NaOH in dil. alc., forming 75% *dibenzylidenecyclopentanone*, m. 188-9° (cf. Vorlander and Hobohm, *Ber.* 29, 1836(1896)). I with Br in CS_2 and with Cl in aq. $CaCO_3$ forms highly unstable halo derivs. The reaction mixt. when hydrolyzed with $CaCO_3$ in H_2O at 60° affords 54% *α-hydroxycyclopentanone* (III), b_{10} 104-8°; *p-nitrophenylhydrazone*, m. 157-8°. II with Br gave an identical III. The latter reacts with semicarbazine, giving *α-cyclobutyl-1,2,4-triazine*, m. 194° (decompn.) (cf. *C. A.* 31, 4281[6]). Oxidation of III with alk. $KMnO_4$ gave glutaric acid. **IX. Isomeric changes of cyclopropylformaldehyde.** E. D. Venus-Danilova and V. F. Kazimirova. *Ibid.* 1438-46.—$CH_2.CH_2.CHCHO$ (I), b. 98-101°, was prepd. by oxidation of cyclopropylcarbinol with CrO_3 and $KHSO_4$ (cf. Dem'yanov and Fortunatov, *C. A.* 2, 1122), and by oxidation of 1-aminomethylcyclopropane with air O in the presence of the Cu catalyst (cf. Shuikina, *C. A.* 31, 5332[6]). The *oxime* of I m. 86°; *semicarbazone*, m. 125-6°. I reacts with 92% H_2SO_4 at 0° to give *α-hydroxybutyraldehyde* (II); *p-nitrophenylosazone*, m. 226°. Oxidation of II with freshly prepd. AgOH gave α-hydroxybutyric acid. II is easily polymerized on standing. Heating I with 60% H_2SO_4 in a CO_2 atm. in a sealed tube at 120-30° for 5 hrs. formed *methylacetylcarbinol*, MeCH(OH)Ac; *oxazone*, m. 166.5-7°; *semicarbazone*, m. 184-5°. I with Br in CS_2 at 0° gave the highly unstable *α,γ-dibromobutyric acid*, identified as the *Et ester*, b_{10} 140-2° (cf. Kizhner, *J. Russ. Phys.-Chem. Soc.* 41, 653(1909)), and as the *α-hydroxybutyrolactone*, b_{10} 138-41°. Approx. 70 references. Chas. Blanc

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BC

a-3

Aldehydes and hydroxyaldehydes of the polymethylene series. IX. Transformations of [illegible]aldehyde. E. D. [illegible] and V. F. [illegible] (J. Geo[illegible] Russ., 1938, [illegible]) [illegible] and 92% H_2SO_4 at [illegible] yield $CH_2CHBr\text{-}CHO$, while with 60% H_2SO_4 at [illegible] the product is $CHMe\text{-}CHMe\text{-}OH$. With Br in CS_2 (?) gives $CH_2Br\text{-}CH_2\text{-}CHBr\text{-}CO_2H$.

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Isomerization of phenylcyclohexylacetaldehyde. R. D. Venus-Danilova and A. I. Bol'shukhin. *J. Gen. Chem.* (U. S. S. R.) 9, 975-84(1939); cf. *C. A.* 32, 2925[1].—Phenylcyclohexylacetaldehyde (2.8 g.) was added by drops to 10 cc. of concd. H_2SO_4 at −5 to −12° within 20 min. The reaction mixt. was poured into 700 g. of ice and then dild. with 150 cc. of water. The products of reaction were extd. with ether. After the usual treatment, the ether was distd. off and the residue was steam-distd. The volatile products were extd. with ether. After removal of the ether, 29% (based on aldehyde used) of *benzyl cyclohexyl ketone* (I) was obtained. However, the main reaction was the formation of dimers and polymers. The above aldehyde (5 g.), on heating with 5 g. of $HgSO_4$ and 12.5 cc. of H_2SO_4 (d. 1.84) in a sealed glass tube in a CO_2 atm. at 128-35°, yielded 60% of I, the rest being complex products of condensation of the aldehyde. For identification purposes the following alcs. and corresponding ketones were prepd.: *benzylcyclohexylcarbinol*, $PhCH_2CH(OH)C_6H_{11}$ (II), m. 58-9°, by the Grignard reaction from $PhCH_2MgCl$ and hexahydrobenzaldehyde, yield 76% (theory); I, b_{10} 163-5°, d_4^{20} 1.0293, d_4^{25} 1.0112 and d_4^{40} 1.0094, n_D^{20} 1.5285, MR_D 61.70, parachor 495.3, prepd. by the oxidation of II with $KMnO_4$ (the yield 60%); semicarbazone, m. 139-40°; oxime, m. 117-18°. *Phenylhexahydrobenzylcarbinol*, $C_6H_{11}CH_2CH(OH)Ph$ (III), b_{10} 156-7°, d_4^{20} 1.0239, d_4^{25} 1.0133 and d_4^{40} 1.0115, n_D^{20} 1.5370, MR_D 63.03, parachor 490.2, prepd. by the Grignard reaction from BzH and $C_6H_{11}CH_2MgI$ in 30% yield; *phenylhexahydrobenzyl ketone*, $C_6H_{11}CH_2COPh$ (IV), b_{10} 161-2°, d_4^{20} 1.0300, d_4^{25} 1.0192 and d_4^{40} 1.0174, n_D^{20} 1.5390, MR_D 61.94, parachor 494.7, prepd. by oxidation of III with $K_2Cr_2O_7$; semicarbazone, m. 192-3°; oxime, m. 99°. On heating 2.5 g. of I with 5.3 g. KOH and 20 cc. alc. in sealed tubes at 145-8° for 6 hrs., 31% of $PhCH_2CO_2H$ and 64% II were obtained. Similar treatment of IV yielded 80% of III (solid), m. 55-7°. The attempt to synthesize phenylcyclohexylacetaldehyde, $Ph(C_6H_{11})CHCHO$, through the acetal failed, giving instead, *sym*-diphenylcyclohexylethane (V), m. 199-201°. For that purpose, *phenylcyclohexylbromomethane*, $Ph(C_6H_{11})CHBr$, m. 41-2°, was prepd. by treating phenylcyclohexylcarbinol, $Ph(C_6H_{11})CHOH$, with PBr_3; the yield was 41.5%. The action of Mg on the methane under the condition of Grignard reaction led to the formation of V. A. A. P.

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In commemoration of I. I. Bergman. E. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 9, 2302-3(1939). Obituary. Gertrude Berend

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Transformation of α-monohalide carbonyl compounds into saturated monobasic acids. E. D. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 11, 817-50(1941).—The author studied the transformation of halogen-substituted ketones into monobasic acids. A satd. soln. of 8[illegible] g. KOH in 200 cc. 96% EtOH was treated dropwise with stirring at room temp. with 95 g. $Me_2CBrCOMe$ in 50 cc. 96% EtOH; after the initial reaction, the mixt. was heated on a steam bath for 1.5 hrs. with stirring; alc. and the neutral products were steam-distd., and the alk. residue was acidified by dil. H_2SO_4 and extd. with Et_2O; the latter on distn. left 19.2 g. Me_2CCO_2H, which was distd. for purification, b. 163-4°, m. 35-5.5°; the yield was 33%. Heating of Bromo ketones with H_2O to boiling in the presence of carbonates yielded only the corresponding keto acids, the acids being formed only in small amts. Thus, $EtCHBrCOPr$ heated with solns. of $BaCO_3$, $CaCO_3$ and K_2CO_3 gave, resp., 50%, 30%, and 20% $EtCH(OH)COPr$ and 1.6%, 9.0% and 10.0% $EtPrCHCO_2H$, b. 208-9°. Boiling $MeCHBrCOEt$ with $BaCO_3$ soln. gave 2.2% $MeEtCHCO_2H$; boiling $Me_2CHCOMe$ with $BaCO_3$ gave 2.0% Me_2CCO_2H. G. M. Kosolapoff

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3RD AND 4TH ORDERS

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0-3

Dehydration of polyethylene alcohols. E. D. Vinos-Danilova and A. I. Bolschakin (J. Gen. Chem. Russ., 1943, 13, 75—86).—cyclohexylcarbinols are readily dehydrated with $H_2C_2O_4$ to the [illegible] derivative of [illegible] or [illegible] but [illegible] not be dehydrated. The [illegible] is [illegible] by the relative ease of dehydrogenation of the [illegible] and dehydrohalogenation of the halohydrates.

α-cyclohexyl-β-phenylethyl alcohol (I) (67 g.) with $H_2C_2O_4$ (386 g.) at 156—170° [illegible] α-phenyl-α-cyclohexylidenethane, $C_{14}H_{18}$ [illegible] 1·6401, d_4^{20} 0·9678, which is [illegible] and adipic acid. (I) is converted by [illegible] $C_6H_5CO_2$ b.p. 160—163°/7 mm., [illegible] Similarly obtained from α-cyclohexyl-β-phenylethyl [illegible] α-phenyl-β-cyclohexylidenethane, b.p. [illegible] 1-β-phenylethyl-cyclohexylene; [illegible] b.p. 145°/58 mm. (Alexandria, [illegible]), from β-cyclohexyl-β-phenylethyl alcohol; and cyclohexylethylene, b.p. [illegible] mm., from cyclohexylmethylcarbinol. [illegible] is not dehydrated by 50% H_2SO_4, [illegible] or $H_2C_2O_4$, and is converted by HBr at 100° into the [illegible] b.p. [illegible] mm. E. A. B.

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Derivatives of 2,4-diphenyl-5,5-dimethyl-2-hydroxy-2,5-dihydrofuran. R. D. Venus-Danilova. *J. Gen. Chem.* (U. S. S. R.) 13, 93-101(in English, 101)(1943).—In the course of investigations of unsatd. compds., Favorskii and Venus reported the prepn. of a compd., at that time assumed to be 2,4-diphenyl-5,5-dimethyl-4-hydroxy-2,5-dihydrofuran (*C. A.* 9, 1471). In the present work the substance was investigated further. It was prepd. according to the previous method. Zerevitinov detns. show it to possess 1 HO group. On the basis of further exptl. work the compd. is shown to be *2,4-diphenyl-5,5-dimethyl-2-hydroxy-2,5-dihydrofuran* (I), which enters some reactions in its tautomeric form: *2-methyl-2,5-diphenyl-2-penten-2-ol-5-one*. The substance (2 g.) in 25 cc. EtOH was treated with 3-4 drops AcOH and heated briefly to reflux; the mixt. was filtered and the EtOH allowed to evap. slowly, yielding 1.9 g. *2-ethoxy analog* of I, m. 97-97.5°; *2-methoxy analog* of I, prepd. analogously, m. 93-3° (from Et_2O); *semicarbazone* of I, m. 169-70° (from 80% EtOH); *phenylhydrazone*, prepd. in 50% AcOH, m. 99-100° (from EtOH); the *oxime* could not be prepd. I treated with excess Br in dry $CHCl_3$ soln. yields I *pentabromide*, bright red solid which readily loses Br on standing in air, or on washing with Et_2O or EtOH; passing dry air through the pentabromide causes loss of Br, leaving the I *tribromide*, m. 168-9° (*2,4-diphenyl-5,5-dimethyl-2-hydroxy-2,3,4-tribromotetrahydrofuran*). The tribromide heated with AcOH dissolves, and on cooling deposits colorless crystals, m. 148-9.5°, apparently *2,4-diphenyl-5,5-dimethyl-2-hydroxy-3-bromo-2,5-dihydrofuran*, m. 149-50° (from benzene-ether); treated with Br, it yields the penta- and tribromides described above; the substance dissolved in EtOH in the presence of a trace of AcOH at the b. p., followed by cooling and standing, gave an *unstd. bromide*, m. 110-11°, while similar treatment with MeOH gives a *monobromide*, m. 104-5°; these 2 substances have an alkoxy group in the 2-position. The tribromide (m. 168-9°), boiled with MeOH, yields *2,4-diphenyl-5,5-dimethyl-2-methoxy-3-bromo-2,5-dihydrofuran*, m. 104-5°; with EtOH, the product is *2,4-diphenyl-5,5-dimethyl-2-ethoxy-3-bromo-2,5-dihydrofuran*, m. 110-11°; the latter, oxidized with CrO_3 in AcOH, yielded Me_2CO and BzOH.

O. M. Kosolapoff

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Isomerization of hydroxy aldehydes. VII. Oxidative-reductive transformation of α-hydroxybutyraldehyde. E. D. Venus-Danilova and V. F. Kazimirova (Leningrad Chem. Tech. Inst.). *J. Gen. Chem.* (U.S.S.R.) **10**, 2099-2108(1946)(in Russian); cf. *C.A.* **32**, 7807[b].—In acid soln. $EtCH(OH)CHO$ (I) is transformed into $MeCH(OH)Ac$ and its oxidation product, Ac_2, which gave in part an aldolization product, *3-methylheptan-3-ol-2,5,6-trione* (II). Some $EtCH(OH)CO_2H$ (III) was also formed. Both in acid media and in the presence of weak alkali with $Cu(OH)_2$ or $Pb(OH)_2$ as catalysts the sole primary product of I is $MeCH(OH)Ac$. I (3 g.), 5 cc. EtOH, and 15 cc. 1% H_2SO_4 failed to react after 5.5 hrs. at 130-5°. I (5 g.), 7 cc. EtOH, and 20 cc. 2% H_2SO_4 gave after 8.5 hrs. at 140-8° 18% $MeCH(OH)Ac$ and 26% Ac_2. I (2 g.), 2 g. $Pb(NO_3)_2$, 5 g. KOH, and 75 cc. H_2O allowed to stand 5.5 months gave 43% polymerization products and 41% III. I (3.5 g.), 1.7 g. $CuSO_4$, 5.3 g. KOH, and 75 cc. H_2O gave after 1.3 hrs. at 100° 14% Ac_2, 37% polymerization products, 23% AcOH, and 12% III. I (4.5 g.), 2 g. $Pb(NO_3)_2$, 5 g. KOH, and 140 cc. H_2O gave after 3 hrs. at 100° 24% $MeCH(OH)Ac$, 20% polymerization products, and 11% III. These transformations are unexplainable by the oxide-intermediate mechanism, which would predict the formation of a primary rather than the actually observed secondary alc. It is possible to explain the reaction by an enolization mechanism: $RCH_2CH(OH)CHO \rightarrow RCH_2C(OH):CHOH \rightarrow RCH:C(OH)CH_2OH \rightarrow RCH(OH)C(OH):CH_2 \rightarrow RCH(OH)COCH_3$. Parabutyraldehyde (426 g.) was bromi-

nated in CS_2 with the theoretical amt. Br to yield 47% crude bromobutyraldehyde, b_{10} 65-8°, which could not be purified by further distn.; it was refluxed with excess $BaCO_3$ until hydrolysis was complete and the filtered soln. was extd. with Et_2O to yield 13 g. *ethylglyoxal*, b_{10} 53-4°, d_4^{20} 1.0404, d_4^{20} 1.0304, d_4^{20} 1.0285, n_D^{20} 1.4178 (*p-nitrophenylhydrazone*, m. 228-30° (from alc.); *disemicarbazone*, m. 226-7°), and crude I, which could not be purified (b_{10} 90-108°). I was finally prepd. in pure state according to Dworzak and Pierri (*C.A.* **23**, 4670), through bromobutyraldehyde acetal, b_9 83°, which on hydrolysis gave 60% I, b_{12} 75-82°, which on standing forms a *dimer*, m. 95°; I gave a *p-nitrophenylhydrazone*, m. 134-5°, and a *p-nitrophenylosazone*, m. 227-8°. The 3rd and 4th isomerization expts. (see above) gave also a small amt. of II as a noncrystallizable oil, which with $PhNHNH_2$ gave an orange-red *pyrazoline* of II, m. 203-4° (from AcOH).

G. M. Kosolapoff

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Lab. Org. Chem., Leningrad Tech. Inst. Ing. Genmet

Isomerization of hydroxy aldehydes. XI. Oxidation-reduction of α-hydroxyenanthaldehyde. E. D. Venus-Danilova and V. F. Kazimirova. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 18, 1816-22(1948); cf. *C.A.* 41, 2006g; 42, 1106.—Enanthaldehyde (I) was polymerized by dry HCl at −20° and the polymer frozen out in 90% yield. The polymer was brominated with cooling and immediately converted to the *bromoenanthaldehyde di-Et acetal*, b_4 100-2° (35%). Hydrolysis of the latter on a steam bath with *N* KOH gave 79% undistd. *α-hydroxyenanthaldehyde* (pure substance, b_4 103-5°; forms on standing a *dimer*, m. 96-100°) (II). Heating 2.2 g. II with 2.5 g. KOH and 1 g. Pb oxide hydrate in 70 ml. H_2O 5 hrs. at 100° gave 56% *butylacetylcarbinol*, b_3 124-5°, 0.9 g. condensation products, including a little *2,5-dibutyl-p-quinone*, m. 144-6° (also obtained by oxidation of the keto alc. by CrO_3), and the *dimer* of the *keto alc.*, b_3 185-9°; the acidic products (28%) contained only *2-hydroxyheptanoic acid*, m. 66°. Use of II dimer in the reaction gave similar results. The isomerization is analogous to that of EtCH(OH)CHO. **XII. Hydroxy ketones with primary alcohol group and their behavior with α-hydroxy aldehydes and isomeric α-hydroxy ketones.** S. N. Danilov and N. S. Tikhomirova. *Ibid.* 1956-62; cf. *C.A.* 25, 1819.—MeEtCO 5, H_2O 2.5, and marble 1, chlorinated at 70° until the marble was consumed, and the reaction mixt. washed gave *EtCOCH₂Cl* (I) (240 g. from 1400 g. EtMeCO), b_{760} 137-8°, b_{110} 83-4°, b_{60} 58-9°, b_{25} 52-3°, b_{17} 40-1°, b_{10} 34-5°, d_4^{20} 1.0850, n_D^{20} 1.4311 (*semicarbazone*, m. 121°); the bulk of the reaction product consists of *MeCHClCOMe*, b. 115-17° (*semicarbazone*, decomp. 128°, hydrolyzed by water, followed by neutralization, to *methylacetylcarbinol semicarbazone*, m. 185-6°). I (60 g.), 70 g. KOAc, and 150 ml. abs. EtOH refluxed 6 hrs. give 42.5% *EtCOCH₂OAc*, b_{10} 80-4°, d_4^{20} 1.0320, n_D^{20} 1.4139 [*semicarbazone*, m. 114° (from EtOH)]; hydrolysis to the alc. is complicated by the need of an inert atm., hence the following procedure is better: 35 g. I, 56 g. dry KO_2CH, and 56 g. MeOH were heated in a sealed tube to 120° and the products (in a CO_2 atm.) distd., yielding 50% *EtCOCH₂OH*, b_{22} 56-7°, b_{23} 85-6°, b_{760} 152-4°, d_4^{20} 1.0186, n_D^{20} 1.4237; in addn. some *ethylglyoxal*, b. 176-8°, is also formed. The alc. does not give a semicarbazone by the usual procedures, but yields *ethylglyoxal disemicarbazone*, m. 228-30°; it does give an *osazone*, m. 115° (from EtOH); *p-nitrophenylhydrazone*, m. 228° (decompn., from EtOH). I heated with excess Ag_2CO_3 in water 6 hrs., followed by distn., gives 2 substances on treatment with *p*-$O_2NC_6H_4$-$NHNH_2$, m. 226-7° (deriv. of $EtCOCH_2OH$, EtCOCHO, or EtCH(OH)CHO) and m. 245° (that of MeAcCHOH). Heating I with $Pb(OH)_2$ in 5% KOH gives 2 isomeric keto alcs.: *methylacetylcarbinol* and $EtCOCH_2OH$; the former is isolated as the *dimer*, m. 95°; the hydrolysis products with KOAc and $H_2NCONHNH_2$.HCl give as a main product a semicarbazone, m. 186-7°, giving no m.p. depression with the deriv. from the ketone obtained by hydrolysis of MeCHClCOMe (deriv. of methylacetylcarbinol). Osazone formation gives *propionylcarbinol osazone*, m. 115°, as well as that of *methylacetylcarbinol*, m. 242°. Hydrolysis of I with $Pb(OH)_2$-KOH 7 hrs. at 150° gives only the MeAcCHOH derivs. (semicarbazone, *p*-nitrophenylhydrazone). G. M. Kosolapoff

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Transformation of pinacols with substituted acetylenic radicals. V. Synthesis and transformations of (diphenylmethyl)(phenylethynyl)ethylene glycol. E. D. Venus-Danilova, E. P. Brichko, and L. A. Pavlova. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 19, 951-8(1949); *A. C.A.* 42, 4166. PhMgBr and Et lactate gave 67% $Ph_2C(OH)CH(OH)Me$, m. 95-6°, which on dehydration gave 2 forms of Ph_2CH L, m. 46° and m. 61°, in 86% total yield. Bromination gave 57% Ph_2CBrAc, m. 55-6° (some product, m. 66°, was concurrently isolated, the hydrolysis of which gave only tars, this product formed largely in the bromination of the high-melting ketone), which on hydrolysis by $BaCO_3$ in water suspension gave 57% $Ph_2C(OH)Ac$, m. 65-6°. This added in Et_2O soln. to PhC:CMgBr at room temp., followed by aq. treatment, gave 51% $Ph_2C(OH)C(OH)(C:CPh)Me$ (I), m. 119-20° (from petr. ether and Et_2O). I (10 g.) and 100 ml. 30% H_2SO_4 boiled 3.5 hrs. gave 4.5 g. starting material and some tar; 36% H_2SO_4 gave a similar result with some indication of a definite transformation, while 38% H_2SO_4 gave from 7.8 g. I an unstated amt. of I and 0.6 g. product, $C_{26}H_{18}O$, m. 100-1°, free of HO groups, and apparently having a ketone structure but giving no CO reactions; its oxidation by $KMnO_4$ in pyridine gave AcPh and BzOH (in 1:2 ratio) only. Hence, the product is $BzCMePh(C:CPh)$. It is also obtained in moderate yields on 2.5 hrs.' refluxing of I with 30% H_2SO_4; concd. H_2SO_4 gave only tar. **VI. Synthesis and transformations of sym. methyldiphenyl(phenylethynyl)ethylene glycol (2,3,5-triphenyl-4-pentyne-2,3-diol).** E. D. Venus-Danilova and L. A. Pavlova. *Ibid.* 1755-65. Addn. of the theoretical amt. of methylbenzoin to PhC:CMgBr in Et_2O and letting stand overnight gave an unpurifiable product, but a 2.5:1 ratio of the RMgBr in 3 days' refluxing in Et_2O gave *2,3,5-triphenyl-4-pentyne-2,3-diol*, m. 36-8° (from Et_2O); attempted distn. *in vacuo* led to decompn. into AcPh and *phenyl(phenylethynyl)carbinol*, b_4 181-3°, m. 49-50° (confirmed by oxidation with CrO_3-AcOH). Boiling the diol (13.5 g.) with 10 vols. 40% H_2SO_4 gave on extn. with Et_2O 6 g. *3,3,5-triphenyl-4-pentyn-2-one*, m. 96-7° (from EtOH), while neutralization of the acidic soln. gave the compd. $MePhC.CPh:CH.CPh(OH).O$ (II), m. 125-7° (from Et_2O-ligroin) (*chloroplatinate*, m. 171-3°, *chloroaurate*, m. 171-6°); heating this hydroxyfuran with EtOH in the presence of AcOH gave the Et ether, a viscous oil. Repetition of the reaction with 30% H_2SO_4 instead of 40% acid gave, from 13 g. diol., 3.3 g. of the above ketone, 2 g. II, and 6.7 g. oil; when 20% H_2SO_4 was used the yield of ketone was 2.2 g. from 23 g. diol, with some 18.5 g. unidentified oil. The mother liquor after isolation of the ketone in all of the above expts. gave a noncrystallizable oil which has an OH group and on attempted distn. yields decompn. products (AcPh and PhCH:CHAc), thus indicating the original structure as $MePhC(OH)CPh:CHBz$, confirmed by oxidation with $KMnO_4$ to AcPh, methylbenzoin, and BzOH, as well as $BzCO_2H$; this keto alc. on heating with 40% H_2SO_4 gave 33.3% II. G. M. K.

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CA

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Transformation of unsymmetric dimethyldiphenylbutynediol in acid media. I. Isomerization of the glycol under the influence of alcoholic sulfuric acid. Yu. S. Zal'kind, E. D. Venus-Danilova, and V. I. Ryabtseva (Leningrad Technol. Inst., Leningrad). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 2222-9(1950).—Heating 10.5 g. $Me_2C(OH)C{:}CC(OH)Ph_2$, m. 116-17°, 80 ml. 95% EtOH, and 7 ml. concd. H_2SO_4 8 hrs. with stirring at 60-70° gave 85.7% *2,2-dimethyl-5,5-diphenyltetrahydro-3-furanone*, m. 66-7°; *semicarbazone*, m. 210° (from EtOH). This, treated with MeMgI, gave 90% *2,2,3-trimethyl-5,5-diphenyltetrahydro-3-furanol*, m. 74-5° (from ligroine), which on oxidation with $KMnO_4$ in H_2O 20 hrs. at 85° gave benzilic acid, AcOH, Ph_2CO, and Me_2CO. If the isomerization is done with 7.6% alc. H_2SO_4 for 6 hrs. at 30-5° there is formed 90% *2-methyl-5,5-diphenyl-3,4-pentanedione*, m. 132-3° (from EtOH); *semicarbazone*, m. 160° (from dil. EtOH); *dioxime*, decomp. 135-41°. The dione with $KMnO_4$ gave Ph_2CO, Me_2CO, and $(CO_2H)_2$, while CrO_3 in AcOH gave Ph_2CH-CO_2H. When 12.5% alc. H_2SO_4 was used in the isomerization, 30% furanone and 70% diketone were formed, while 15.1% H_2SO_4 gave an 85:15 division of the products. Possible courses of the reaction are discussed. II. Condensation of the glycol with ethanol in the presence of sulfuric acid. E. D. Venus-Danilova and V. I. Ryabtseva. *Ibid.* 2230-5.—Stirring 10 g. $Me_2C(OH)C{:}CC(OH)Ph_2$, 80 ml. EtOH, and 0.2 ml. concd. H_2SO_4 at room temp., then at 30-5° for 20 hrs., neutralization, and filtration, gave after standing 10 hrs. a little of the above described diketone, m. 132°, while the evapd. mother liquor, on treatment with petr. ether, gave a mechanically separable mixt. of the diketone and 75% of a new substance, m. 64-5°, identified as *2,2-dimethyl-5,5-diphenyl-3-ethoxy-2,5-dihydrofuran*, which, stirred 2 days with $KMnO_4$ in H_2O at 70-80°, gave *2,3-dimethyl-5,5-diphenyl-2,5-pentanediol-3,4-dione*, m. 83-4°, and some Me_2CO. This product (0.35 g.), refluxed 2 hrs. with 30 ml. EtOH and 7 ml. concd. H_2SO_4, gave a red oil, which could not be crystd., but apparently was *2,2-dimethyl-5,5-diphenyltetrahydro-3,4-furandione*, as treatment with o-$C_6H_4(NH_2)_2$ gave the corresponding *quinoxaline*, m. 161-2° (from EtOH). Among the acidic products of the above oxidation was $Ph_2C(OH)COCO_2H$, an oil, characterized by its *Ag salt* and *semicarbazone*, decomp. 135°. Stirring 2,2-dimethyl-5,5-diphenyl-3-ethoxy-2,5-dihydrofuran (1 g.) 3 hrs. with 8 ml. EtOH, 0.7 ml. concd. H_2SO_4, and 0.5 ml. H_2O at 70-5° gave 2,2-dimethyl-5,5-diphenyltetrahydro-3-furanone, identical with that described in the previous abstr.

G. M. Kosolapoff

1951

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CA

The transformation of unsymmetric dimethyldiphenyl butynediol in acid media. I. The isomerization of the glycol under the influence of an alcoholic sulfuric acid. Yu. S. Zal'kind, E. D. Venus-Danilova, and V. I. Ryabtseva. *J. Gen. Chem.-U.S.S.R.* 20, 2300-16(1950)(Engl. translation). II. The condensation of the glycol with ethyl alcohol in the presence of sulfuric acid. E. D. Venus-Danilova and V. I. Ryabtseva. *Ibid.* 2317-22. See C. 1 45, 7075a/b. B. L. M.

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Isomerization of methylphenylethynylcarbinol in acid medium. E. D. Venus-Danilova, A. P. Ivanov, and I. I. Martynov (Lensovet Technol. Inst., Leningrad). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 1808-10(1951).—Treatment of $(BrMgCl)_2$ simultaneously with PhAc and EtOH in Et_2O gave 31% *MePhC(OH)C:CH* (I), m. 48.5-9.5°; purification is best done by steam distn. of the petr. ether ext. of the reaction mixt. after hydrolysis. A 36-42% yield of corresponding diol is also obtained. I (4 g.) in 20 ml. 60% AcOH and 3.5 g. $Hg(OAc)_2$ in 30 ml. 60% AcOH kept 2 hrs. on a steam bath, gave 2.2 g. unchanged I and a trace of a reducing substance. However, 4 g. I in 20 ml. EtOH, 2 g. HgO, and 100 ml. 1:7 H_2SO_4 kept 3 hrs. on a steam bath gave from several runs an av. yield of 28.8% *methylphenylacetylcarbinol* (3-hydroxy-3-phenyl-2-butanone), b_5 90-8° (*semicarbazone*, m. 182-5°), and 14.8% *MePhC:CHCHO*, b_5 76-7° (*semicarbazone*, m. 198-9°); the best sepn. is via the semicarbazone route. Oxidation of the aldehyde by Ag_2O failed, yielding only AcPh. The aldehyde probably forms by anionotropic rearrangement of the acetylene-allene type through the enolic form of the aldehyde. G. M. Kroshapoff

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CA

Transformations of pinacolones with substituted acetylenic radicals VIII Synthesis and transformations of trimethyl(phenylethynyl)ethylene glycol (2,3-dimethyl-5-phenyl-4-pentyn-2,3-diol). K. D. Venus-Danilova, V. I. Serkina, and L. A. Pavlova (Leningrad Technol. Inst., Leningrad). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 2110-10(1951); cf. C.A. 44, 5854f, 6400d, 7291g.—MeAcCOH and PhC:CMgBr gave 52% *2,3-dimethyl-5-phenyl-4-pentyn-2,3-diol*, b. 150-2°; this (12 g.) refluxed 3 hrs. in 10 parts 30% H_2SO_4 gave Me_2CO and a mixt. of *2,3-dimethyl-5-phenyl-5-penten-2-ol-4-one* (I) and its dehydration product, *2,3-dimethyl-5-phenyl-1,3-pentadien-5-one* (II). Direct sepn. of I failed and the mixt. was dehydrated by hot Ac_2O, giving the fairly pure II, b. 80-90° (*semicarbazone*, m. 132°); oxidation with $KMnO_4$ gave Ac, HCO_2H, BzOH, and $BzCO_2H$. In addn. to I and II, the acid treatment of the diol also gave a small amt. of *2,3-dimethyl-5-phenyl-4-pentyn-2-one*, b. 120-8°, which failed to yield a semicarbazone; oxidation with $KMnO_4$ gave Me_2CO, BzOH and $Me_2C(OH)CO_2H$, and traces of HCO_2H and AcOH, as well as possibly $CH_2:CHMeCO_2H$ (obtained only as polymer). In addn., an unstated amt. of C_6H_5O, b. 112-21°, was isolated and provisionally identified as *α-ethylidenacetophenone*. Fairly pure I, b. 120-30°. Me groups at the 1st C atom at the pinacol and the acetylene-allene rearrangement, while Me at the 2nd C atom aids the pinacol rearrangement.

G. M. Kosolapoff

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

A

Hydroxydihydrofurans. I. Oxidation and reduction of 2,4-diphenyl-5,5-dimethyl-2-hydroxy-2,5-dihydrofuran. M. D. Venus-Danilova and A. N. Orlova (Leningrad Chem. Tech. Inst., Leningrad). *Zhur. Obshchei Khim.* (*J. Gen. Chem.*) 22, 333-9(1952).—2,4-Diphenyl-5,5-dimethyl-2-hydroxy-2,5-dihydrofuran (I) (cf. Paverskii and Venus, *C.A.* 9, 1471) (2 g.) treated with 3.2 g. $KMnO_4$ in 2% soln. 1.5 hrs. at 40° gave 1 g. unchanged material, m. 100-1°, no neutral oxidation products except Me_2CO, and a trace of HCOOH, as well as appreciable amts. of EtOH and EtCOOH, m. 54°. Hydrogenation of I in EtOH with Pt black at room temp. gave much tar and some 10% *2,4-diphenyl-5,5-dimethyl-2,5-dihydrofuran*, m. 135-7°, and 50% *2,4-diphenyl-5,5-dimethyltetrahydrofuran* (II), b_4 158-60°. If the hydrogenation is stopped after the uptake of 4 H atoms, it is noted that the reaction rate is sharply reduced after addn. of 2 atoms of H and the main product is II. Hydrogenation over colloidal Pd-in-EtOH gave after the uptake of 2 H atoms the same tar as above, while uptake of 4 atoms gave II.

G. M. Kosolapoff

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E.D. VENUS*DANILOVA, V.M. ALIBITSKAYA

May 52

USSR/Chemistry - Acetylene Derivatives

"Conversion of Secondary-Tertiary Glycols of the Acetylene Series by Means of Salts, I. 1,2,4,-Triphenyl-Butyy-3-Diol-1,2," Lab of Org. Chem, Leningrad Technological Inst. im Lenosovet

Zhur Obshch Khim, Vol 22, No5, pp 816-821

The action of mercuric chloride on diphenyl-phenylacetylenyl-ethylene glycol at 100° results in a 88.6% yield of 2,3,5-triphenyl furane.

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VENUS-DANILOVA, E. D.

Chem

Chemical Abst.
Vol. 48 No. 5
Mar. 10, 1954
Organic Chemistry

Transformations of secondary-tertiary diols of the acetylene series under the influence of mercury salts. I. 1,3,4-Triphenyl-3-butyne-1,2-diol. E. D. Venus-Danilova and V. M. Al'bitskaya (Lensoviet Inst. Technol., Leningrad). *J. Gen. Chem. U.S.S.R.* 22, 879-82(1952)(Engl. translation).—See *C.A.* 47, 3266c. H. L. H.

AF 7-27

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENUS-DANILOVA, E. D.

Venus-Danilova, E. D., Serkova, V. I.- "Investigation of transformations of pinacones with subsituted acetylene radicals. Part 9. Synthesis and transformations of triphenyl-phenylacetylenylethylene glycol (1, 1, 2, 4-tetraphenylbutyne-3-diol-1,2)". (p. 1563)

SO: Journal of General Chemistry, (Zhurnal Obshchei Khimii), 1952, Vol. 22, No. 9

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

1. ZAL'KIND, Yu. S.; VENUS-ANILOVA, Ye. D.; MIKHAYLOVA, V. N.

2. USSR (600)

4. Diphenylbutynediol

7. Synthesis and properties of ethers of disecondary glycols. Part 1. Preparation of methyl and ethyl ethers of diphenylbutynediol. Zhur.ob.khim. 22 no. 10 1952.

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

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENUS-DANILOVA, Ye.D.; AL'BITSKAYA, V.M.

Transformations of secondary-tertiary acetylenic α-glycols under the action of salts of mercury. II. 3-Methyl-5-phenyl-4-pentyn-2,3-diol. Zhur. Obshchey Khim. 22, 1568-72 '52. (MLRA 5:9)
(CA 47 no.17:8683 '53)

1. Lensovet Technol. Inst., Leningrad.

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENUS-DANILOVA, E. D.

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

Transformations of secondary-tertiary acetylenic α-[illegible] under the action of salts of mercury. II. 3-Methyl-5-phenyl-4-pentyn-2,3-diol. P. D. Venus-Danilova and V. M. Al'bitskaya. J. Gen. Chem. (U.S.S.R.) 22, 1611-1 (1952) (Engl. translation). - See C.A. 47, 8683f.
H. L. H.

11-5-51

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENUS-DANILOVA, E. D.

Chemical Abst.
Vol. 48 No. [illegible]
Apr. 25, 1954
Organic Chemistry

Synthesis and properties of ethers of disecondary [illegible] glycols. I. Preparation of methyl and ethyl ethers of diphenylbutynediol. Vu. S. [illegible] E. D. Venus-Danilova, and V. S. Mikhailova. J. Gen. Chem. (U.S.S.R.) 22, [illegible] (Engl. translation). See C.A. 47, 6921c.
H. L. H.

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VENUS-DANILOVA, E.D.; GOBELIK, M.V.

Synthesis and conversion of acetylene alcohols containing polymethylene rings. Part 1. 1-phenylacetylenyl-cyclopentanol-1. Zhur.ob.khim. 23 no. 7:1139-1142 Jl '53. (MLRA 6:7)

1. Laboratoriya organicheskoy khimii Leningradskogo tekhnologicheskogo instituta imeni Lensoveta. (Alcohols)

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ZAL'KIND, Yu.S.; VENUS-DANILOVA, E.D.; MIKHAYLOVA, V.N.

Synthesis and properties of ethers of di-secondary γ-glycols. Zhur.ob. khim. 23 no.7:1143-1145 Jl '53. (MLRA 6:7)

1. Laboratoriya organicheskoy khimii Leningradskogo tekhnologicheskogo instituta imeni Lensoveta. (Glycols) (Ethers)

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acid.

Author

Title

Periodical

Abstract

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

Zhur. Ob. Khim., [illegible], Ed. [illegible], [illegible] [illegible] [illegible] [illegible] [illegible] [illegible]

Card [illegible]

Abstract [illegible]
1909. Graph.

Institution : The Lensoviet Technological Institute, Leningrad

Submitted : September 29, 1953

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENUS- DANILOVA, E. D.

USSR/ Chemistry Isomerization

Card : 1/1 Pub. [illegible]

Authors : Venus-Danilova, E. D., Ryabtseva, V. I., and Frigoryeva, L. A.

Title : Conversion of nonsymmetrical dimethyl-diphenyl-butinediol in an acid medium, [illegible]

Periodical : Zhur. ob. khim. [illegible], [illegible], August, 1954

Abstract : Experiments showed that the substance with melting point of 132 - 133°, formed as result of isomeric conversion of nonsymmetrical dimethyl-diphenyl-butinediol under the effect of an aqueous sulfuric acid medium [illegible]

Institution : The Lensoviet Technological Institute, Leningrad

Submitted : February 16, 1954

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"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

VENUS-DANILOVA, E.D.; FABRITSY, A.; ORLOVA, A.N.

Study of oxydihydrofurans. Part 5. Basic properties of 5,5-dimethyl-2-tert-butyl-4-phenyl-2-oxydihydrofuran-2,5. Zhur.ob.khim. 26 no.4:1160-1165 Ap '56. (MLRA 9:8)

(Furan)

APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1"

"APPROVED FOR RELEASE: 09/01/2001 CIA-RDP86-00513R001859420001-1

USSR/Organic Chemistry. Synthetic Organic Chemistry. E-2

Abs Jour: Ref Zhur - Khimiya, No. 8, 1957, 26883.

Author : Venus-Danilova, E.D., Fabritsy, A.
Inst :
Title : Study of Conversions of Pinacols with Replaced Acetylene Radicals. XI. Action of Mercury Chloride and Bromide on Asymmetrical Dimethyl-phenyl-Acetylenyl-Ethylenglycol.

Orig Pub: Zh. obshch. khimii, 1956, 26, No. 6, 1609 - 1616.

Abstract: The following compounds were obtained by boiling equimolar amounts of 2-methyl-3,5-diphenylpentine-4-diol-2,3 (I) and $HgCl_2$ in alcohol solution 1 to 6 hours: 5,5-dimethyl-2,4-diphenyl-2-oxydihydrofuran-2,5 (II), melting point 160-161° (decomposes), the mercury complex thereof

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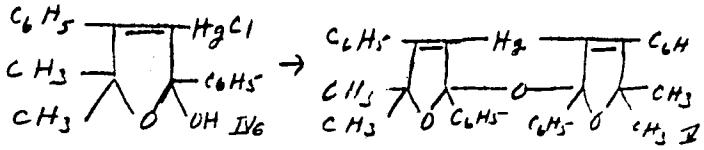
USSR/Organic Chemistry. Synthetic Organic Chemistry. E-2

Abs Jour: Ref Zhur - Khimiya, No. 8, 1957, 26883.

(III) and the mercury-organic compound thereof (IV) (which can exist in two forms IVa and IVb) separated by the action of NH_3 on the mixture of IV and II as a product of symmetrization (V) (melting point 203-204°) (decomposes).

IVa ⟶



The yield of III rises to 93% at the interaction of I with HgCl in presence of concentrated HCl, which indicates the parallel course of two reactions: conversion of I into II and formation of IV with separation of HCl, which participates

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USSR/Organic Chemistry. Synthetic Organic Chemistry. E-2

Abs Jour: Ref Zhur - Khimiya, No. 8, 1957, 26883.

together with II in the composition of III. The presence of II in the reaction mixture means that the isomerization of I into II proceeds, as it seems, more rapidly than the interaction of I with $HgCl_2$, though the mechanism of II formation has not been made clear. It has been established that II does not react with $HgCl_2$, and that I is converted into II in presence of 20%-ual HCl, yield 52%. The mercury complex $C_{18}H_{17}OHgBr_3.H_2O$ (melting point 180-183°) described by the author earlier (see RZhKhim, 1956, 71562) was produced by the interaction of I with $HgBr_2$ (yield 84%). It is surmised that IV forms from I and $HgCl_2$ according to the scheme: $I+HgCl_2 \rightarrow (CH_3)_2C(OH)C(C_6H_5)-(OH)C(HgCl)=CClC_6H_5 \rightarrow (CH_3)_2C(OH)(C_6H_5)=C(HgCl)-$

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AUTHORS: Venus-Danilova, E. D.; Serkova, V. I.; Yel'tsov, A. V. 79-2-13/58

TITLE: Study of Pinacol Conversions with Substituted Acetylene Radicals, Part 14. Synthesis and Conversions of Nonsymm. Methyl-Diphenyl-Tertiary-Butylacetylenyl-Ethylene Glycol (1,1-Diphenyl-2,5,5-Trimethylhexine-3-diol-1,2) (Issledovaniye prevrashcheniy pinakonov s zameshchennymi atsetilenovymi radikalami. XIV. Sintez i prevrashcheniye nesimm.metil-difenil-tretichnobutilatsetilenil-etilenglikolya (1,1-difenil-2,5,5-trimetil-geksin-3-diol-1,2).

PERIODICAL: Zhurnal Obshchey Khimii, 1957, vol 27, No 2, pp. 334-339 (U.S.S.R.)

ABSTRACT: The authors synthesized a new representative of acetylene series pinacols of nonsymmetrical methyl-diphenyl-tertiary-butylacetylenyl-ethyleneglycol and showed that this pinacol when heated with a 41.5% aqueous solution of sulfuric acid converts into enin alcohol - 1,1-diphenyl-2-methylene-5.5 dimethylhexine-3-01-1. The effects of different sulfuric acid concentrations on nonsymmetrical ethylene glycols are described. The structure of the enin alcohol - 1.1-diphenyl-2-methylene-5,5-dimethylhexine-3-01-1 was

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Study of Pinacol Conversions with Substituted Acetylene Radicals. Part 14.

proven during its oxidation and derivation of benzophenone, benzilic, formic and trimethylacetic acids. It was revealed that the reaction of the alcohol solution of nonsymmetrical methyl-diphenyl-tertiary-butylacetylenyl-ethylene glycol with 2,4-dinitrophenylhydrazine in the presence of sulfuric acid leads to the formation of 2,4-dinitro-phenylhydrazone, corresponding to isomeric ethylene gamma-katoalcohol - 1,1-diphenyl-2,5,5-trimethylehexene-2-01-1-on-4.
There are 19 references, of which 17 are Slavic

ASSOCIATION: Leningrad Technological Institute imeni Leningrad Soviet

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VENUS-DANILOVA, E.D.; PAVLOVA, L.A.; FAERITSY, A.

Study of oxydihydrofurans. Part 6: Reaction of 5,5-dimethyl-2,4-diphenyl -2-oxydihydrofurna-2,5 and 2,4-dinitrophenylhydrazine. Zhur. ob. khim. 27 no.9:2423-2429 S '57. (MIRA 11:3)

1. Leningradskiy tekhnologicheskiy institut imeni Lensoveta.
(Furan) (Hydrazine)

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AUTHORS: Pavlova, L. A., Fabritsy A. Venus-Danilova, E. D.

79-28 3-19/61

TITLE: Investigation of the Reactions of Pinacols With Substituted Acetylene Radicals (Issledovaniye prevrashcheniy pinakonov s zameshchennymi atsetilenovymi radikalami). XV. The Action of Sulfate of Mercury on the Asymmetric Dimethylphenyl-Phenyl-Acetylenyl- and the Asymmetric Methyl-Diphenyl-Phenylacetylenyl-Ethylene-Glycols (XV. Deystviye sernokisloy rtuti na nesimm. dimetil- fenil -fenil_atsetilenil- i nesimm. metil difenil-fenilatsetilenil -etilen_glikoli)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 3, pp. 651-657 (USSR)

ABSTRACT: Using mercury salts the authors have not succeeded until now in obtaining hydration products of acetylene-double-tertiary α-glycols. It was observed that the asymmetric methyl-diphenyl-phenylacetylenyl-ethylene glycol (formula I b) in aqueous as well as in acetone solution converts to the earlier described $C_{46}H_{36}O_2$ (ref 3) in the presence

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Investigation of the Reactions of Pinacols With Substituted Acetylene Radicals. 79-28-3-19/61

XV. The Action of Sulfate of Mercury on the Asymmetric Dimethylphenyl--Phenyl-Acetylenyl- and the Asymmetric Methyl-Diphenyl Phenylacetylenyl-Ethylene-Glycols

of small amounts of sulfuric acid. It can be assumed from the dehydration and the regrouping within the molecule that in the beginning a 2,5,5-triphenyl 4-vinyl-dihydro=furfuran-4,5 is formed which then converts to a dimer, analogous to 2,3,3,4-tetramethyl-2-(β,β) dimethylvinyl-dihydrofurfuran-2,3 (ref. 4). The parallel experiment to convert glycol (I b) with diluted sulfuric acid without sulfate of mercury was not successful, as was to be expec=ted. The asymmetric dimethyl-phenyl-phenylacetylenyl-ethy=lene glycol (I a) was subjected to the action of sulfate of mercury on various conditions. In aqueous and weakly acidous sulfuric acid 5,5-dimethyl (-2,4-diphenyl-2 oxydihy=drofurfuran-2,5 (II) resulted, in acetone solution a vis=cous oil which did not have any hydroxyl group and slowly decolored a bromo- and potassiumpermanganate solution. The synthetized product could be obtained pure by vacuum distil=lation. The same product resulted from the action of sul=fate of mercury on glycol in acetone solution. According to its elementary solution and its molecular weight this

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