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EXCERPTA MEDICA Sec 9 Vol 13/10 Surgery Oct. 59

6210. PRIMARY CIRCULAR SUTURE OF THE ARTERIES IN INFECTED WOUNDS (Russian text) - Sichenikov I. A. - KHIRURGIYA 1958, 6 (98-106) Tables 1 Illus. 3

Experiments were carried out on 74 dogs; 62 experiments with administration of antibiotics and 12 without antibiotics. Arteries were sutured mechanically in 35 cases and manually in 39. Primary circular suture in suppurating wounds gave favourable results in the first group. Best results were obtained if surgical treatment of infected wounds was carried out within 24 hr. after their infliction. The results depend to a great extent on the rational choice of antibiotics, based on analysis of the wound microflora. Mechanical suture has considerable advantages in infected wounds over the manual suture. The results of these experiments make it possible to recommend wider application of primary suture of arteries, even in conditions with pronounced inflammation.

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Akademiya nauk SSSR. Sovet po izucheniyu proizvoditel'nykh sil

Chernaya metallurgiya (Ferrous Metallurgy) Moscow, Izd-vo AN SSSR, 1960. 275 p. (Series: Razvitiye proizvoditel'nykh sil Vostochnoy Sibiri) Errata slip inserted. 2,000 copies printed.

Ed.: G.I. Lyudogovskiy, Candidate of Technical Sciences; Ed. of Publishing House: G.M. Makovskiy; Tech. Ed.: Ye.V. Makuni; Editorial Board of this vol.: S.S. Dvorin, S.V. Orchininskiy, Candidate of Technical Sciences, A.N. Pokhvisnev, Doctor of Technical Sciences, and A.A. Fedotov, Engineer; Editorial Board of Series: I.P. Bardin,(Deceased), Academician, Academy of Sciences USSR (Chief. Ed.); M.A. Lavrent'yev, Academician, Academy of Sciences USSR; S.I. Vol'fkovich, Academician, Academy of Sciences USSR; V.I. Dikushin, Academician, Academy of Sciences USSR; V.S. Nemchinov, Academician, Academy of Sciences USSR; V.I. Veyts, Corresponding Member, Academy of Sciences USSR; G.D. Levitskiy, Corresponding Member, Academy of Sciences USSR; N.N. Nekrasov, Corresponding Member, Academy of Sciences USSR; L.B. Pustovalov, Corresponding Member, Academy of Sciences USSR; T.S. Khachaturov, Corresponding Member, Academy of Sciences USSR; N.P. Rostovtsev, Academician, All-Union Academy of Agricultural Science; A.N. Popov, Member, Academy of Building and Architecture USSR; L. Ye. Grafov, Deputy Chairman, State Planning Committee of the Council of Ministers RSFSR; A.D. Gusbev, Member, State Planning Committee of the Council of Ministers RSFSR; A.Ye. Probst, Professor; V.F. Vasyutin, Professor; V.A. Krotov, Professor; P.V. Vasil'yev, Doctor of Economics; G.I. Lyudogovskiy, Candidate of Technical Sciences; P.A. Letunov, Candidate of Geology and Mineralogy; and M.G. Shkol'nikov, Candidate of Economics.

PURPOSE: This collection of papers is intended to furnish information on industrial resources in Eastern Siberia and to provide a basis for future developmental planning in the field of ferrous metallurgy.

COVERAGE: The collection is a summary of the proceedings of the Ferrous Metallurgy Section of the Joint Conference of Representatives of the Academy of Sciences USSR, the State Planning Commission, and the Council of Ministers RSFSR on the Development of the Industrial Resources of Eastern Siberia. The collection deals with four main areas of development in Eastern Siberia: 1) Mineral resources, 2) the fuel base, 3) prospects for the development of ferrous metallurgy, and 4) problems in the development of electrometallurgy. A list of the 112 members of the Section with their affiliations is given in the Appendix. References accompany several of the articles.

Sichenko, V.K. Prospects for the Development of Coke and Chemical Production From Coking Coals of Eastern Siberia 95

Panchenko, S.I. Raw Material Base for Coke Supply for Eastern Siberian Ferrous Metallurgy 104

Sapozhnikov, L.M. Continuous Coking and SemiCoking of Eastern Siberian Coals 110

Pokhvisnev, A.N. and B.M. Zlobinskiy. Coke From Eastern Siberian Brown Coals 114

Review of Adresses on Reports Dealing With the Development of the Coke and Coke Byproduct Coke Industry of Eastern Siberia 121

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SICHENKO, V.K.; IVANOV, B.V.; POLYAKOV, I.I.; REZNIKOV, A.A.; DORFMAN, G.A.; IZRAELIT, E.M.; NOTYCH, A.G.; TOPYGIN, L.A.; CHALYY, G.Ya.; STETSENKO, Ye.Ya.; UDOVICHENKO, L.V.; FILIPPOV, B.S., nauchn. red.; LERNER, R.Z., nauchn. red.; GOL'DIN, Ya.A., glav. red.; KULESHOV, M.M., red.; POLOTSK, S.M., red.

[By-product coke industry] Koksokhimicheskoe proizvodstvo. Moskva, Metallurgiia, 1965. 167 p. (MIRA 18:7)

1. TSentral'nyy nauchno-issledovatel'skiy institut informatsii i tekhniko-ekonomicheskikh issledovaniy chernoy metallurgii. 2. Direktor TSentral'nogo nauchno-issledovatel'skogo instituta informatsii i tekhniko-ekonomicheskikh issledovaniy chernoy metallurgii (for Kuleshov).

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SICHER, J.

A

Synthesis of pseudoconhydrine. I. Synthesis of one isomeride of racemic 2-propyl-5-hydroxypiperidine. F. Šorm and J. Sicher. *Collection Czechoslov. Chem. Communs.* 14, 331-44(1949)(in English). Oxidation of 2-alkyl-8-hydroxyquinoline with fuming HNO_3 in the presence of V_2O_5 gives 2-alkylquinolinic acids from which 5-substituted 2-alkylpyridines and piperidines can be prepd. Alkylation of 8-methoxyquinoline with PrI gives 2-propyl-8-methoxyquinoline (I), $b_{0.5}$ 116-24° (picrate, m. 152-3°). Refluxing with 49% HBr formed 2-propyl-8-hydroxyquinoline (II), $b_{0.5}$ 89-91° (picrate, m. 155-6°; benzoate, m. 73-4°). Oxidation gave 41% 2-propyl-5,6-pyridinedicarboxylic acid (III), m. 142-3°, and 2-propyl-6-pyridone-5-carboxylic acid, m. 161°. Decarboxylation in boiling $EtCO_2H$ gave 2-propyl-5-pyridinecarboxylic acid, m. 129-30°, which was converted via the Et ester, b_{12} 135-6°, hydrazide, m. 90-1°, azide, and urethan, m. 70°, to 2-propyl-5-aminopyridine, b_{11} 134-6° (picrate, m. 161°). Diazotization yielded 2-propyl-5-hydroxypyridine, m. 91-2°, which was hydrogenated (Pt catalyst) to *dl*-2-propyl-5-hydroxypiperidine, m. 90-1°. Oxidation of 2-propylquinoline (IV) with 10% H_2O_2 in H_2SO_4 and $CuSO_4$ formed 2,3,4-pyridinetricarboxylic acid and not the expected III. II can be obtained from IV via the sulfonic acid in poor yields.

John Howe Scott

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CA SICHER, J.

10

Comments on the condensation of p-nitrobenzoyl chloride with malonic and cyanoacetic. M. Svoboda, J. Sicher, J. Gut, and F. Hrabák (Tech. Univ., Prague, Czech.). *Chem. Listy* 46, 51-2(1952).—An ether soln. (100 ml.) contg. 9.23 g. p-$O_2NC_6H_4COCl$ (I), 8 g. $CH_2(CO_2Et)_2$, and 1.15 g. Na in 10 ml. EtOH was allowed to stand 4 days at room temp., then acidified with dil. H_2SO_4, treated with Na_2CO_3 soln., and evapd.; a compd., m. 94-5° (from MeOH), suggested to be $(p\text{-}O_2NC_6H_4CO)_2C(CO_2Et)_2$ or perhaps $(p\text{-}O_2NC_6H_4CO)(p\text{-}CO_2C_6H_4CO_2)C(CO_2Et)_2$ was obtained in 2.96-g. yield. Acidifying the Na_2CO_3 ext. pptd. 2.45 g. p-$O_2NC_6H_4COCH(CO_2Et)_2$, m. 43-5°. Similar reaction occurred between I 6 g. and Na cyanoacetate (from 3.3 g. of the ester) and gave 4 g. $(p\text{-}O_2NC_6H_4CO)_2C(CN)CO_2Me$, m. 153-4° (from MeOH), and 1.7 g. p-$O_2NC_6H_4COCH(CN)CO_2Me$, m. 157° (from MeOH), also obtained in 35% yield from $EtOMgCH(CN)CO_2Me$ and I.

M. Hudlický

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SICHEK, V.

SICHER, J.; FARKAS, J.; SORM, F.

Studies in the chloramphenicol series. Part 4. Synthesis of 1-(p-nitrophenyl)-2-hydroxymethyl-2-dichloro-acetamido-1,3-propandiol and a correction [in English with summary in Russian]. Sbor.Chekh.khim.rab. 18 no.1:102-105 F '53. (MLRA 7:6)

1. Chentral Chemical Research Institute, Prague.
(Chloramphenicol)

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Sicher, Jiri

Chloramphenicol series. V. Analogs containing chlorine in the side chain and oxazolines. Jiří Farkaš and Jiří Sicher (Czech. akad. ved., Prague). *Collection Czechoslov. Chem. Communs.* 18, 460-80(1953)(in English).—See *C.A.* 49, 218*b*. VI. Steric course of the reduction of dehydrochloramphenicol and related compounds. Jiří Sicher, Miroslav Svoboda, Magdalena Hrdá, Josef Rudinger, and František Šorm (Czech. akad. ved., Prague). *Ibid.* 487-99.—See *C.A.* 49, 219*b*. H. L. H.

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Sicher, J., Farkas, J. "Studies in the chloramphenical series." V. Analogues containing chlorine in the side chain and oxazolines. p. 552 CASOPIS PRO PESTOVANI MATEMATIKY. CZECHOSLOVAK MATHEMATICAL JOURNAL. Vol. 47, no. 4, Apr. 1953, Praha, Czechoslovakia.

SO: Monthly List of East European Accessions, LC., Vol. 3, No. 1, Jan. 1954, Uncl.

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Sicher, J. and others "Studies in the chloramphenicol series." VI. The steric course of the reduction of dehydrochloramphenicol and of related compounds. p. 565 CASOPIS PRO PESTOVANI MATEMATIKY. CZECHOSLOVAK MATHEMATICAL JOURNAL. Vol. 47, no. 4, Apr. 1953, Praha, Czechoslovakia

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SICHER, J.

SICHER, J.; SVOBODA, M.; FARKAS, J.; SORM, F.

Studies in the chloramphenicol series. Part 7. The side reactions in the reduction of dehydrochloramphenicol [in English with summary in Russian]. Sbor.Chekh.khim.rab. 19 no.2:317-329 Ap '54. (MLRA 7:6)

1. Department of Organic Synthesis, Institute of Organic Chemistry, Czechoslovak Akademy of Science, Prague. (Chloromycetin)

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ISHER, J.; [illegible], J.; [illegible], A.

"Studies on the Chloramphenicol Series. VIII. Some Unsaturated Derivatives. In English." p. 545,
(COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SBORNIK CHEKHOSLOVATSKIKH KHIMICHESKIKH RABOT, Vol. 19, No. 3, June 1954, Praha, Czechoslovakia)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4
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Sicher, Jiri

CZECH

Studies in the chloramphenicol series. IX. Side-chain mercapto analogs and thiazolines. III. Fatty and [illegible] Sicher (Czech. Akad. Ved, Prague). *Chem. Listy* 48, 695-[illegible] 1954. *Collect. Czechoslov. Chem. Commun.* 19, 7[illegible]-[illegible] (in English). Includes [illegible] *Ref.* 44, 5[illegible]h.—The prepn. of the mercaptoanalogs of chloramphenicol, *threo*-(I) and *erythro*-*p*-$O_2NC_6H_4CH(OH)CH(NHCOCHCl_2)CH_2SH$ (II), is described and the results of pharmacol. tests of the chloro and mercapto analogs of chloramphenicol given. Adding AcSH (6.69 g.) to a soln. of 12.12 g. *p*-$O_2NC_6H_4COC(NHCOCHCl_2)CH_2$ (III) in 20 ml. C_6H_6, filtering the crystals, washing with ether, and crystg. from C_6H_6 gave 13.36 g. *p*-$O_2NC_6H_4COCH(NHCOCHCl_2)CH_2SAc$ (IV), m. 132° (from C_6H_6). Similarly, 1.51 g. III in 2 ml. dioxane treated with 0.8 g. BzSH at 50° gave 1.4 g. *p*-$O_2NC_6H_4COCH(NHCOCHCl_2)CH_2SBz$ (V), m. 184° (from dioxane). In the same manner was prepd. *p*-$O_2NC_6H_4COCH(NHAc)CH_2SAc$, m. 140-1° (from EtOH), in 72% yield. Distg. the mixt. of 3.79 g. IV, 70 ml. iso-PrOH, and 4.08 g. Al (iso-$PrO)_3$ 5 hrs. under CO_2, decompg. the residue with H_2O 5 min. at 80°, extg. with Et_2O, distg. off the solvent, and crystg. the residue from EtOH yielded 2.03 g. (64%) *erythro*-*p*-$O_2NC_6H_4CH(OH)CH_2N{:}C(CHCl_2)S.CH_2$ (VI), m. 132°. The same compd. was obtained in 10% yield by the Meerwein-Ponndorf reduction of V. Refluxing VI (1.28 g.) in C_6H_6 (5 ml.) with 1.52 g. *p*-$MeC_6H_4SO_2Cl$ and 2 ml. Me_3N 2 hrs., dilg. the mixt. with 20 ml. Et_2O, filtering off the salt, evapg. the solvents, and adding petr. ether to the residue gave 0.55 cryst. *erythro*-*p*-$O_2NC_6H_4CH(OSO_2C_6H_4Me)CH_2N{:}C(CHCl_2)S.CH_2$, m. 138° (from EtOH).

Heating 35.7 g. V with 33 g. Al (iso-$PrO)_3$ 2 hrs. with 480 ml. C_6H_6, decompg. the residue after distg. off the solvent with a soln. of 10 g. tartaric acid in 100 ml. H_2O, heating the

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mixt. 10 min. at 50°, dilg. with [illegible] ml. H_2O, extg. with AcOEt, and distg. off the AcOEt gave 8.07 g. (22%) *erythro*-*p*-$O_2NC_6H_4CH(OH)CH(NHCOCHCl_2)CH_2SBz$ (VII), m. 207° (from EtOH). Using fresh liquid Al(iso-PrO)$_3$, increased the yield to 49%. VI (1.6 g.) refluxed 15 min. with 200 ml. 10% AcOH yielded 1.28 g. (1.06 g. after crystn. from H_2O) II, m. 136.7° (from PhMe). II was recyclized to VI by heating with Al(iso-PrO)$_3$ in iso-PrOH (31 mg. out of 70 mg.). Treating 0.34 g. II in 70 ml. EtOH contg. 1 ml. C_5H_5N with 0.15 g. BzCl dissolved in 20 ml. EtOH 30 min. at 15° gave 0.17 g. VII, m. 207°. *threo*-*p*-$O_2NC_6H_4CH(OH)CH_2N{:}C(CHCl_2)OCH_2$ (*C.A.* 43, 3011*c*) (VIII) (1.52 g.) [illegible]

in 5 ml. dioxane added to Et_2O satd. with HBr at −15°, dild. after 5 min. with 15 ml. AcOH, the soln. washed with three 30-ml. portions H_2O, dried, and evapd. yielded 0.82 g. *threo*-*p*-$O_2NC_6H_4CH(OH)CH(NHCOCHCl_2)CH_2Br$ (IX), m. 146° (from 50% EtOH). Treating a soln. of 0.75 g. IX in 10 ml. Me_2CO with a soln. contg. 0.26 g. BzSK in 10 ml. Me_2CO yielded 0.21 g. *threo*-*p*-$O_2NC_6H_4CH(OH)CH(NHCOCHCl_2)CH_2SBz$, m. 164° (from EtOH). Acetylation of IX with Ac_2O and H_2SO_4 gave 87% *threo*-*p*-$O_2NC_6H_4CHOAcCH(NHCOCHCl_2)CH_2Br$ (X), m. 178–9° (from PhMe). Refluxing 8 g. X in C_6H_6 340 ml. with 5.5 g. P_2S_5 3 hrs. gave, after chromatographic purification, 1.84 g. (27%) *threo*-*p*-$O_2NC_6H_4CH(OAc)CH_2N{:}C(CHCl_2)SCH_2$ (XI), m. 102.5° [illegible]

(from EtOH). Refluxing XI (0.54 g.) [illegible] ml. EtOH with 1.21 g. Al(iso-PrO)$_3$ 3 hrs., distg. off the EtOH, heating the residue with 5 ml. 10 min. at 90° [illegible] g. off the [illegible] and [illegible] with Et_2O yield [illegible] *threo*-*p*-$O_2NC_6H_4$-

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$CH(OH)CH.N:C(CHCl_2).S.CH_2$ (XII), m. 131° (from C_6H_6). Heating the mixt. of 0.32 g. XII, 10 ml. AcOH, and 18 ml. H_2O 30 min. on the steam bath under N_2, dilg. the soln. with 2.1 ml. H_2O, filtering the turbid soln. with C_6, and evapg. the filtrate *in vacuo* gave 0.08 g. I, m. 195°. Heating 0.30 g. XI 5 min. at 100° in 15 ml. 50% HCO_2H and dilg. the mixt. with H_2O gave 0.25 g. (0.10 g. after crystn.) *threo-p*-$O_2NC_6H_4CH(OAc)CH(NHCOCHCl_2)CH_2SH$, m. 155° (from C_6H_6). Refluxing 8.07 g. VII with 35 ml. $SOCl_2$ 1.5 hrs., distg. off the $SOCl_2$ *in vacuo*, and crystg. the residue from C_6H_6 gave 5.54 g. *erythro-p*-$O_2NC_6H_4CHClCH(NHCOCHCl_2)CH_2SBz$, (XIII), m. 132°. Refluxing 5.35 g. XIII with 50 ml. BuOH 4 hrs. and distg. off the BuOH yielded 1.25 g. *threo-p*-$O_2NC_6H_4CH(OH)CH(NHCOCHCl_2)CH_2SBz$, m. 164° (from EtOH). Refluxing 4.45 g. *erythro-p*-$O_2NC_6H_4CHClCH(NHCOCHCl_2)CH_2OBz$ (XIV) 30 min. in 20 ml. dry collidine (170°) and filtering off the collidine hydrochloride deposited after the addn. of Et_2O yielded 2.9 g. *trans-p*-$O_2NC_6H_4CH.O.C(CHCl_2):N.CHCH_2OBz$, m. 122–4° (from EtOH). Re-

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CZECH

Terpenes. LXIII. Total synthesis of chamazulene. A simple general synthesis of 1,4,7-substituted azulenes. Jiří Novák, František Šorm, and Jiří Sicher (Czech. Akad. věd, Prague). *Chem. Listy* 48, 1648–85(1954); *Collection Czechoslov. Chem. Communs.* 19, 1264–73(1954)(in English); cf. *C.A.* 49, 9064*e*.—A general synthesis of *1,4,7-trialkylazulenes* has been worked out based on the hydrogenation of a suitably substituted *ar*-dihydroxybenzosuberene to the satd. diol, oxidation of the diol to the coresponding dicarboxylic acid, and cyclization to the desired azulene skeleton. *Chamazulene* was synthesized in this way. 2,3-$(MeO)_2C_6H_3COCl$ and Pr_2Cd gave 72% 2,3-$(MeO)_2C_6H_3$-COPr (I), $b_{0.2}$ 104°; *semicarbazone*, m. 154.5° (from MeOH). Refluxing 120 g. I, 61 g. $Me_2NH.HCl$, 35 g. $(CH_2O)_n$, and 2.5 ml. concd. HCl in 450 ml. EtOH 4 hrs. with efficient stirring, adding 10 g. $(CH_2O)_n$, refluxing 8 more hrs., distg. off most of the EtOH, stirring the cryst. residue several times with Et_2O, dissolving the crystals in H_2O, liberating the bases with 400 ml. 10% NaOH, extg. the mixt. with Et_2O, concg. the ext. *in vacuo* to 80 ml., and treating the concentrate with dry HCl gave 66 g. 2,3-$(MeO)_2C_6H_3CO$-$CHEtCH_2NMe_2.HCl$ (II), m. 141° (from EtOH), and 65 g. recovered I. Treating 21.5 g. II in 60 ml. H_2O with 40 ml. 10% NaOH, extg. the liberated base with Et_2O, adding with cooling 20 g. MeI, evapg. the excess MeI and Et_2O *in vacuo*, stirring the crystals with 100 ml. EtOH, treating the mixt. with $NaCH(CO_2Et)_2$ [prepd. from 2.2 g. Na and 15 g. $CH_2(CO_2Et)_2$] in 100 ml. EtOH, refluxing the mixt. until no more Me_3N escaped (8 hrs.), distg. off the EtOH, dissolving the NaI in H_2O, refluxing the crude diester 2,3-$(MeO)_2C_6H_3COCHEtCH_2CH(CO_2Et)_2$ with 40 ml. 10% NaOH 2 hrs., acidifying the mixt., decarboxylating the free acid by heating 30 min. at 170°, and distg. the crude acid *in vacuo* gave 15.6 g. 2,3-$(MeO)_2C_6H_3COCHEtCH_2CH_2$-$CH_2H$ (III), $b_{0.4}$ 190°. Hydrogenation of 23.7 g. III in 200 ml. MeOH over 3 g. 5% Pd-C at 80–90° gave 20.6 g. 2,3-$(MeO)_2C_6H_3CHEtCH_2CH_2CH_2CO_2H$ (IV), $b_{0.4}$ 181°. Cyclizing 23 g. IV by heating 1 hr. at 70° with 750 g. PPA

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Sicher, Jiri

Stereochemical studies. Introductory remarks. Jiří Sicher (Czech. Akad. věd, Prague). *Chem. Listy* 49, 1318-20; *Collection Czechoslov. Chem. Communs.* 20, 1389-90 (1955)(in Engl.).—A program for the following series of papers on stereochemistry of noncyclic compounds is displayed, and the fundamental terms are explained. I. Steric course of the acid-catalyzed cyclization of *N*-thiobenzoyl derivatives of 1,2-amino alcohols. A new stereospecific reaction. Jiří Farkaš and Jiří Sicher. *Chem. Listy*. 49, 1320-9; *Collection Czechoslov. Chem. Communs.* 20, 1391-1401(1955)(in English).—A new stereospecific reaction was found in the acid-catalyzed cyclization of *N*-thiobenzoyl derivs. of epimeric 1,2-amino alcs. The *threo*-epimers give exclusively *trans*-2-oxazolines, the *erythro*-epimers yield only 20-40% *cis*-2-oxazolines whereas the main reaction products are *trans*-2-thiazolines. The starting $RCH(OH)CH(NH_2)R'$ (I) were prepd. according to the literature. Physical consts. are given for: R = R' = Me, *threo*, m. 18-19°, n_D^{20} 1.4450; *erythro*, m. 42-3°; R = Ph, R' = CO_2Et, *threo*, m. 81-2°; *erythro*, m. 84°; R = R' = Ph, *threo*, m. 126-8°; *erythro*, m. 103°. Hydrogenating 7 g. *erythro*-compd. R = Me, R' = Ph in 99% EtOH (210 ml.) contg. 0.2% ethanolic HCl, over 0.7 g. PtO_2 at 15°, extg. the alkalized mixt. with ether, and treating the base with excess HCl in EtOH gave 3.1 g. I.HCl (R = Me, R' = C_6H_{11}) m. 189-90°. The thiobenzoyl derivs. of the amino alcs., $RCH(OH)CHR'NHCSPh$, were prepd. in 3 ways: method *A*: 0.01 mole of the amino alc. in 10 ml. C_5H_5N was heated 1 hr. at 100° with 0.011 mole $PhCSSCH_2CO_2H$, the mixt. dild. with 100 ml. Et_2O, the ether layer extd. with 5% HCl, with a satd. soln. of $NaHCO_3$, the ether distd. off, and the residue stirred with petr. ether and recrystd. Method *B*: A soln. of 0.01 mole $PhSSCH_2CO_2H$ in 15 ml. 2*N* NaOH was added to 0.01 mole of the amino alc. or its HCl salt, the mixt. allowed to stand 5 hrs. at 20°, the thiobenzoyl deriv. filtered off and crystd. Method *C*: a 2-oxazoline deriv. (0.02 mole) was added to 25 ml. of a soln. of $(NH_4)_2S$ in MeOH, the mixt. allowed to stand 10 days, dild. with 500 ml. H_2O, extd. with Et_2O, the Et_2O evapd., the residue distd. with C_6H_6 and crystd. The compds. of the general formula $RCH(OH)CHR'NHCSPh$ are given (R, R', configuration, method of prepn., yield in %, and m.p.): Me, Me, *threo* (Ia), *B*, 42, 80-1° (from C_6H_6-petr. ether); Me, Me, *erythro* (II), *B*, 54, 85° (from C_6H_6-petr. ether); Ph, Me, *threo* (III), *A*, *C*, 23% and 50%, resp., 79-80° (from C_6H_6-petr. ether); Ph, Me, *erythro* (IV), *A*, 67, 136° (from C_6H_6) [*$PhCSSCH_2CO_2H$ salt* of *erythro*-*$PhCH(OH)CHMeNH_2$*, m. 141-2°]; Me, Ph, *threo* (V), *C*, 32, 115-16° (from EtOH); Me, Ph, *erythro* (VI), *A*, 30, 132° (from EtOH); Ph, CO_2Et, *threo* (VII), *A*, 46, 89-90° (from C_6H_6-petr. ether); Ph, CO_2Et, *erythro* (VIII), *A*, 61, 141-4° (from EtOH); Ph, Ph, *threo* (IX), *C*, 65, 158° (from EtOH) [the reaction requires 10 days for completion; otherwise, an addn. compd. of the oxazoline and the product (1:1), m. 118-20°, is isolated]; Ph, Ph, *erythro* (X), *A*, 48, 134-5° (from EtOH); cyclohexyl, Me, *erythro* (XI), *A*, 77, 141° (from 80% EtOH); Me, cyclohexyl, *erythro* (XII), *A*, 45, 92° (from C_6H_6-petr. ether). Cyclization of Ia-XII was carried out as follows: 0.003 mole Ia-XII was heated with 5 ml. 10% HCl in EtOH in a sealed tube at 100° until the yellow color disappeared. Two thirds of the EtOH was distd. off at 15 mm., and the residue was treated with Et_2O. With III, V, VII, IX, and VI, cryst. $RCH(OBz)CHR'NH_3Cl$ (XIII) deposited, the mother liquors contg. the corresponding thiazolines were alkalized with aq. NH_3, extd. with petr. ether, the petr. ether evapd., and the residual thiazoline distd. From the aq. layer after the extn. of the thiazolines with petr. ether, small amts. of $RCH(OH)CHR'NHBz$ crystd. on standing. Transformation of the *threo* derivs. to XIII (compd., hrs. of reaction, yield in %, m.p. of XIII): III; 2, 62, 198-200° (from EtOH-H_2O); back acyl

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migration gave *threo-PhCH(OH)CHMeNHBz*, m. 127°; V: 2, 57, 190–1° (from 5% HCl), back acyl migration gave *threo-MeCH(OH)CHPhNHBz*, m. 147° (from EtOH); VII: 2, 41, 190–1° (from 5% HCl); IX: 2, 73, 210° (from EtOH-H_2O); I gave *trans*-MeCH.CHMe.N:CPh.O (XIV) HCl salt, 1 hr., 64%, m. 133–4° (from EtOH-Et_2O). The cyclization of the *erythro* derivs. gave RCH.CHR'.N:CPh.S (XIVa) (compd., time of heating, yield in %, b.p. of XIVa, m.p. of the picrates): II, 1, 62, b_{15} 155–8°, 133° (from MeOH); IV, 5, 67, $b_{0.65}$ 162–7°, 141° (from EtOH); VI, 3, 50, $b_{0.2}$ 135–40°, 144° (from MeOH); in addn., 24% of *erythro-MeCH(OBz)CHPhNH₃Cl*, m. 189–90° (from 5% HCl), was isolated; VIII, 5, 55, m. 65–7° (from petr. ether), $b_{0.2}$ 175°, —; X, 2, 78, m. 93° (from petr. ether), —, —; XI, 1, 63, $b_{0.2}$ 125–30°, 147–8° (from MeOH); XII, 1, 55, $b_{0.2}$ 130°, m. 169° (from MeOH); MeCH(OH)CHMeNH₂ and BzCl gave *erythro-MeCH(OH)CHMeNHBz* (XV), m. 122°. Treating 1 g. XV at 0° with 3 ml. $SOCl_2$, distg. off the $SOCl_2$ after standing 1 hr. at 15°, and crystg. the residue from EtOH-Et_2O gave 0.95 g. *HCl salt* of XIV, m. 133–4° (from EtOH-Et_2O); *free base* XIV, b_{15} 120°; *picrate*, m. 148° (from EtOH). Treating 7.85 g. XI in 20 ml. C_6H_6 with a soln. of 2.2 g. NaOH in 20 ml. H_2O and a soln. of 7.7 g. BzCl in 15 ml. C_6H_6 at 15° gave 11.2 g. *erythro-C_6H_{11}CH(OH)CHMeNHBz* (XVI), m. 126° (from C_6H_6). Treating at 5° 11.2 g. XVI with 30 ml. $SOCl_2$, letting the soln. stand 3 hrs. at 15°, distg. off at 15 mm. the $SOCl_2$, treating the residue with 50 ml. H_2O, neutralizing the soln. with 20% NaOH, extg. the oxazoline deriv. with Et_2O, and distg. the ext., yielded 8.9 g. *trans-C_6H_{11}CH.CHMe.N:CPh.O*, $b_{0.15}$ 135–7°; *picrate*, m. 171° (from EtOH). **II. Reaction of *N*-acyl derivatives of 1,2-amino thiols in acidic solution.** Jiří Sicher and Miroslav Svoboda. *Chem. Listy* 49, 1330–5; *Collection Czechoslov. Chem. Communs.* 20, 1403–8(1955)(in English).—The epimeric *threo*- and *erythro*-RCH(SAc)-CHR'NHCOR" (I) behave differently in acidic medium. After the formation of the common intermediate, a 2-hydroxythiazolidine deriv., the *threo*-epimers give exclusively derivs. of *trans*-2-thiazoline (IIa) whereas the *erythro*-epimers give, in addn. to *cis*-2-thiazoline (IIb), RCH(SCOR"CHR'NH₂.HCl (III). The *N*-acetyl derivs. of I give substantially larger amts. of III than the Bz derivs. The different behavior is explained as follows: the formation of IIa from *trans*-2-hydroxythiazoline is not sterically impeded since the introduction of the double bond does not influence the mutual distance of R and R' in the *threo*-epimers. On the other hand, in *cis*-2-hydroxythiazoline formed from the *erythro*-epimers, the introduction of the double bond results in increasing the steric interaction of R and R' which are brought closer to each other. The steric factors will, therefore, interfere with the formation of IIb. I (R" = Ph) gain, after the formation of IIa and IIb, a conjugation of the formed double bond with the aromatic ring. I (R" = Me) do not have the possibility of having such a conjugated system, and therefore, the acyl migration resulting in III will prevail over the formation of IIa and IIb. Heating a soln. of *threo*-PhCH(SAc)CHMeNH₂Bz (460 mg.) in EtOH satd. with HCl 8 hrs. at 100° in a sealed tube, evapg. the soln. to dryness, dilg. with H_2O, extg. with Et_2O, and chromatographing the residue after the evapn. of the Et_2O gave 320 ml. (benzene fraction) of *trans-PhCH.CHMe.-*

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N:CPh.S; *picrate*, m. 141–2° (from EtOH). Heating a soln. of 3.13 g. *erythro*-PhCH(SAc)CHMeNHBz in EtOH (10 ml.) contg. HCl 8 hrs. at 100°, filtering off 180 mg. of *erythro-PhCH(SBz)CHMeNH₂.HCl*, m. 190–2° (from EtOH-Et_2O), evapg. the mother liquors to dryness, dilg. the residue with H_2O, extg. the soln. with Et_2O, and distg. the ext. gave 1.35 g. *cis*-2,5-diphenyl-4-methyl-2-thiazoline, $b_{0.4}$ 145°; *picrate*, m. 123–4° (from EtOH). Heating 1.38 g. *threo*-PhCH(SAc)CHPhNHBz 1 hr. at 100° with 50 ml. EtOH satd. with HCl, distg. off the EtOH, adding 20 ml. H_2O, extg. the mixt. with Et_2O, evapg. the solvent, and chromatographing the residue gave 1.53 g. crude and 1.44 g. cryst. *trans*-2,4,5-triphenyl-2-thiazoline (IIIa), m. 93–4°, giving isomorphous crystals, m. 68–9°. Heating 11.5 g. *erythro*-PhCH(SAc)CHPhNHBz with 200 ml. EtOH satd. with HCl 6 hrs. on the steam-bath, distg. off the EtOH, adding 50 ml. H_2O and 100 ml. Et_2O, filtering off the crystals (1.68 g.) of *erythro-PhCH(SBz)CHPhNH₂.HCl*, m. 228° (from EtOH-Et_2O), extg. the mother liquor with Et_2O, evapg. the Et_2O, and chromatographing the residue gave 6.29 g. crude and 5 g. cryst. *cis* isomer of (IIIa), m. 84–5°. Heating 3.3 g. *threo*-PhCH(SAc)CH(CO_2Et)NHBz with 100 ml. EtOH satd. with HCl 5 hrs. on the steam-bath, distg. off the EtOH, dilg. the residue with H_2O, and extg. the soln. with Et_2O gave 2.9 g. of an oil which yielded on boiling with petr. ether 150 g. insol. *trans*-2,5-diphenyl-4-carboxy-2-thiazoline, m. 137–8° (from C_6H_6), and 1.62 g. sol. *trans*-2,5-diphenyl-4-carbethoxy-2-thiazoline (IV), m. 65–6° (from petr. ether). Similar treatment of 1.65 g. *erythro*-PhCH(SAc)CH(CO_2Et)NHBz gave 350 mg. *cis* isomer (V) of IV, m. 93–4° (from Et_2O), and IV (from mother liquors after treatment with petr. ether). Heating 19 hrs. gave IV exclusively. IV was obtained by refluxing 1 hr. 0.52 g. *threo*-PhCH(SH)CH(NH_2)CO_2Et with 0.3 g. PhC(OEt):NH in 5 ml. $CHCl_3$, dilg. the mixt. with 20 ml. Et_2O and extg. 3 times with a small vol. H_2O. Heating 240 mg. *threo*-PhCH(SAc)CHPh(NHAc) in 10 ml. EtOH satd. with HCl 4 hrs. on the steam-bath gave 570 mg. *trans*-2-methyl-4,5-diphenyl-2-thiazoline (VI), $b_{0.15}$ 132°. Similar treatment of *erythro*-PhCH(SAc)CHPhNHAc gave 34.5% *cis* isomer of VI, m. 67–8° (from petr. ether). Infrared spectra of IV and V and of the corresponding oxazolines are given. III. Steric course of the reaction of *N*-acyl derivatives of 1,2-amino alcohols with thionyl chloride. Jiří Sicher and Magdalena Pinková. *Chem. Listy* 49, 1336–50; *Collection Czechoslov. Chem. Communs.* 20, 1409–25(1955)(in English).—1,2-*N*-acylamino alcohols react with $SOCl_2$ according to the configuration to give either 2-oxazoline (with inversion), or a β-chloro amide (without inversion). *erythro*-PhCH(OH)CHMeNHCO$C_6H_4NO_2$-*p* (I) gave, after a short action of $SOCl_2$ 2-(*p*-nitrophenyl)-4-methyl-5-phenyl-2-oxazoline (II) and on prolonged action of $SOCl_2$ by a secondary reaction *erythro-PhCHClCHMeNH-CO$C_6H_4NO_2$-p* (III). *threo*-PhCH(OH)CHMeNHCOC_6H_4-NO_2-*p* (IV) yielded by the reaction with $SOCl_2$ 30% *threo-PhCHClCHMeNHC$_6H_4NO_2$-p*(V) and probably III, hydrolysis of which gave 30% *threo-PhCH(OCO$C_6H_4NO_2$-p)CHMeNH₂.HCl* (VI). III and V were not formed by secondary hydrolysis of the corresponding 2-oxazolines since these are stable under the conditions used for the reaction with $SOCl_2$. The direct hydrolysis of the reaction mixt. after the action of $SOCl_2$ upon IV yielded a mixt. of *erythro*- and *threo*-amino alcohols with the ratio of *threo* to *erythro* = 3:2. The two following couples, *erythro*- and *threo*-MeCH-

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(OH)CHPhNHCOC$_6$H$_4$NO$_2$-*p* (VII, VIII), and *erythro*- and *threo*-C$_6$H$_{11}$CH(OH)CHMeNHC$_6$H$_4$NO$_2$-*p* (IX, X) were transformed, with inversion, to *trans*- and *cis*-2-(*p*-nitrophenyl)-4-phenyl-5-methyl-2-oxazoline (XI, XII), and *trans*- and *cis*-2-(*p*-nitrophenyl)-4-methyl-5-cyclohexyl-2-oxazoline (XIII, XIV). The different result of the reaction of $SOCl_2$ with IV is ascribed to the polar effect of the Ph group since the corresponding cyclohexyl deriv. X behaved normally. Hydrolysis of the β-chloro amides afforded the corresponding derivs. RCH(O$_2$CC$_6$H$_4$NO$_2$-*p*)CHR'NH$_2$.HCl having the inverted configuration. The amino alcohols were prepd. from the appropriate isonitroso ketones by hydrogenation over Raney Ni, or by the Meerwein-Ponndorf reduction of *p*-nitrobenzamido ketones during which formation of a 2-oxazoline deriv. was observed. The *threo*-epimers were obtained from the *erythro*-epimers by the reaction with $SOCl_2$ followed by hydrolysis. The cyclohexyl derivs. were prepd. from the Ph deriv. by hydrogenation over PtO_2. *erythro*-PhCH(NH$_2$)CHMeOH was transformed to *trans*-β-methylstyrene oxide (XIVa) (infrared spectra of cis and trans epoxides are given). The 2-oxazolines of cis and trans configuration were prepd. for identification purposes from *p*-nitrobenzamino ethers and the amino alcohols. Hydrogenation of 407 g. BzCMe:NOH in 7.5 l. EtOH over 160 g. Raney Ni at room temp. and 120-50 atm. gave 150 g. *erythro*-*Ph(OH)CHMeNH$_2$* (XV), m. 104-5°. Refluxing 11.2 g. BzCHMeNH$_2$.HCl with 12 g. *p*-O$_2$NC$_6$H$_4$COCl (XVI) in 150 ml. PhMe 6 hrs., distg. off the PhMe, and crystg. the residue from EtOH gave 11 g. *BzCHMeNHCOC$_6$H$_4$-NO$_2$-p* (XVII), m. 123-4°. Treating 37 g. HCl salt of XV in 300 ml. H_2O with 16 g. NaOH in 200 ml. H_2O and 33 g. XVI in 500 ml. C_6H_6 50 min. gave 55 g. crude and 50 g. pure I, m. 189-90° (from EtOH). The same compd. was prepd. by refluxing 2.5 hrs. 9 g. XVII with 9.2 g. (iso-PrO)$_3$Al and 200 ml. iso-PrOH, by decomp. the residue after the evapn. of the solvent with 50 ml. H_2O, by evapg. the mixt. and extg. the residue with AcOEt, in a yield of 8 g. Dissolving 4.5 g. I in 15 ml. $SOCl_2$, evapg. the soln., dissolving the residue in 50% EtOH in the presence of 8 ml. 6*N* HCl in EtOH, refluxing the soln. 5-10 min., and filtering with C gave on cooling 4 g. *HCl salt* of IV, m. 215°; IV, (by alkalization in a 58% yield), m. 170° (from 75% EtOH). Refluxing 1.5 g. IV with 10% HCl 8-10 hrs., extg. the soln. with Et_2O, evapg. the aq. soln. to dryness, and crystg. from EtOH and Et_2O gave 0.7 g. *threo-PhCH(OH)CHMeNH$_2$-HCl*, m. 169-70°; *free base* (in 66-7% yield from the HCl salt in Et_2O with dry NH_3), m. 71° (from C_6H_6). Hydrogenation of PhC(:NOH)Ac over Raney Ni gave 53% *erythro-PhCH(NH$_2$)CHMeOH* (XVIII), m. 85°. Heating 2.2 g. PhCHAcNH$_2$.HCl with 2.4 g. XVI in 40 ml. PhMe 4-5 hrs. gave 2.8 g. *PhCHAcNHCOC$_6$H$_4$NO$_2$-p* (XIX), m. 156-7°. XVIII and XVI gave 80% *erythro-PhCH(NHCOC$_6$H$_4$NO$_2$-p)-CHMeOH* (XX), m. 154-5°. The same compd. was prepd. by the Meerwein-Ponndorf reduction of XIX in a yield of 68%; 6.6% of XII, m. 125°, was isolated as a by-product. The reaction of XX with $SOCl_2$ and the subsequent hydrolysis gave 62% of the *HCl salt* of *threo-PhCH(NHCOC$_6$H$_4$-NO$_2$-p)CHMeOH* (XXI), m. 207-8°; *free base* XXI, m. 153-4°. Hydrolysis of XXI gave 55% *threo-PhCH(NH$_2$)CHMeOH*, m. 90.5-1.5°. The *erythro*-configuration of XVIII was proved by prepg. *PhCH(CHMeOH)NMe$_3$I*, m. 176° (yield 56%) and by transforming it to XIVa. Hydrogenation of 10.5 g. of XV.HCl in 300 ml. 99% EtOH over 1 g. PtO_2 in the presence of 0.3 ml. 99% EtOH satd. at 0° with HCl (room temp., normal pressure) gave 8 g. *erythro-C$_6$H$_{11}$-CH(OH)CHMeNH$_2$*, m. 82-3° (XXII). XXII and XVI gave 53% *erythro-C$_6$H$_{11}$CH(OH)CHMeNHCOC$_6$H$_4$NO$_2$-p*, m. 163-4° (from 75% EtOH). Similarly was prepd., by hydrogenating the *threo*-PhCH(OH)CHMeNH$_2$.HCl, *threo-C$_6$H$_{11}$CH(OH)CHMeNH$_2$* (yield 93%), m. 83-4° (from petr. ether). Nitrobenzoylation gave 82% of a crude compd. contg. di-*p*-nitrobenzoyl deriv. It was treated

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with 100 ml. 1% HCl in EtOH 3 days), the monoacyl deriv. was thus transformed to the HCl salt, which yielded 66% *threo*-$C_6H_5CH(OH)CHMeNH_2COC_6H_4NO_2$-*p* (XXIII), m. 120-1° (from 50% EtOH). The same compd. was prepd. by heating XIII with concd. HCl by way of its HCl salt, m. 200-1° (from EtOH-Et_2O). The reaction of the *p*-nitrobenzoyl derivs. of the amino alcohols was carried out by dissolving the compd. in a min. amt. of $SOCl_2$ (1-2 ml. per 1 g. of the compd.) at 18-25°, by treating the mixt. after a certain time with ice and AcOEt, by evapg. the AcOEt ext., and by crystg. the residue, contg. 10-15% of the HCl salt of the *O*-acyl deriv. Thus were prepd. the following (starting acyl deriv., time of action of $SOCl_2$ (in min.), the product, m.p., yield, and the yield of the comparative compd. prepd. by heating on the steam-bath equimol. amts. of the amino alcohol and Et *p*-nitrobenzimidate given): I, 1, II, 77-8°, 78%, 64%; VII, 1, XI, 125°, 77%, 81%; VIII, 30, XII, 121-2°, 53%, 50%; IX, 120, XIII, 113-14°, 60%, 75%; X, 240, XIV, 124-5°, 67%, 72%. Dissolving 1.5 g. IV in 5 ml. $SOCl_2$, decompg. the mixt. after 1 min. with ice, extg. with C_6H_6 gave 31% V, 128-9° (from C_6H_6). The mother liquors contained 30% VI, m. 215° (from H_2O). In another expt., the residue after the evapn. of C_6H_6 from the ext. (2.85 g.) was refluxed 6 hrs. with 80 ml. and 2 ml. 3N HCl, the soln. evapd. to dryness, the residue extd. with hot C_6H_6 gave 0.2 g. *erythro*- and *threo*-amides, and undissolved 2.08 g. of the *threo*- and *erythro*-$PhCH(O_2CC_6H_4NO_2\text{-}p)CHMeNH_2.HCl$ which were transformed by acyl migration to 1.13 g. I, m. 189-90° (from EtOH), and 0.74 g. VI. *erythro*-$PhCH(O_2CC_6H_4NO_2\text{-}p)CHMeNH_2.HCl$, m. 210°, was obtained in a 9% yield by treating I min. cis isomer (XXIV) of II with $SOCl_2$, decompg. the mixt. with ice and extg. with AcOEt; 89% of XXIV was recovered, m. 110-1°. Transformation of the 2-oxazolines to β-chloroacyl amides was effected by passing dry HCl into a cooled dioxane suspension of the oxazoline, by heating the mixt. until all of the HCl salt dissolved, and by evapg. the soln. (method A), or by removing the solvent from the HCl salt pptd. and by heating the residue 3-5 min. at 190-60° (method B), or by using in method B H_2O instead of dioxane (method C). Thus were prepd. the following β-chloroacyl amides, method of prepn., yield in %, m.p. (from C_6H_6), and R, R', % yield and m.p. of $p\text{-}O_2NC_6H_4CO_2CHRCHR'\text{-}NH_2.HCl$ obtained by refluxing the chloracyl amide 1.5 hrs. on the steam-bath in 75% EtOH: III, A, 60, 132-3°, Ph, Me, *threo*, 95, 210°; V, B, 57, 128-9°, Ph, Me, *erythro*, 28, 212°; *erythro*-$MeCHClCHPhNHCOC_6H_4NO_2\text{-}p$, B, 35, 156-7°, Me, Ph, *threo*, 62, 198°; *threo*-$MeCHClCHPhNHCOC_6H_4NO_2\text{-}p$, B, 49, 166-7°, Me, Ph, *erythro*, 33, 202°; *erythro*-$C_6H_{11}CHClCHMeNHCOC_6H_4NO_2\text{-}p$, C, 30, 140°, C_6H_{11}, Me, *threo*, 55, 190°; *threo*-$C_6H_{11}CHClCHMeNHCOC_6H_4NO_2\text{-}p$, C, 75%, 140-1°; recovered 100% after the hydrolysis with 75% EtOH. IV. Reaction of 2-oxazolines with thioacetic acid. New syntheses of *vic*-amino thiols. Miroslav Svoboda, Jiří Sicher, Jiří Farkaš, and Magdalena Pánková. *Chem. Listy* 49, 1351-62; *Collection Czechoslov. Chem. Communs.* 20, 1426-38(1955)(in English).—Epimeric 2-oxazolines of the general formula RCH.CHR'.N:CR'.O react with AcSH to give RCH(SAc)CHR'NHCOR'. The reaction is not general and fails with certain derivs. The 2-oxazolines having Ph in position 5 show different reactivity for the trans and cis isomers, the latter being much less reactive. *cis*-2-Phenyl-4,5-trimethylene-2-oxazoline (I) (*cis*-aminocyclopentanol) gave normal *S-acetyl-N-benzoyl-cis-2-aminocyclopentanethiol* (II), whereas the oxazolines derived from *cis*- and *trans*-aminocyclohexanol, *cis*- and *trans-2-phenyl-4,5-tetramethylene-2-oxazolines* (III, IV) reacted with AcSH differently and gave *N-acetyl-o-thionobenzoyl-cis*- (and *trans*-)*2-aminocyclohexanol* (V, VI) which were transformed by means of Ag_2O

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to *cis*- and *trans*-benzoyl analogs (VII, VIII) of V and V. The cis deriv. III gave the normal product *S-acetyl-N-benzoyl-cis-2-aminocyclohexanethiol* (IX) in addn. to V. The different behavior is explained by different distortion of the normal cyclic conformations by the annelation with the 5-membered oxazoline ring. This annelation makes the C—O bond almost inaccessible in the case of IV. The diacylamino thiols which could not be prepd. by the above procedure from the oxazolines were prepd. by the reaction of AcSH with ethyleneimines. During the prepn. of oxazolines from the amino alcohols by the reaction with Et benzimidate, the *threo* epimers showed much smoother reaction course than the *erythro* derivs. The 2-oxazolines were prepd. as follows: 0.01 mole of the amino alcohol was refluxed with 0.011 mole of Et benzimidate 4–8 hrs. on the steam-bath (in some cases 5–8 hrs. at 120–30°), the unreacted imidate was distd. off, and the residue was crystd. or subjected to chromatography or distn. (method A). *erythro*-PhCH(OH)CHPhNBz (4.35 g.) was added to conc. H_2SO_4 (20 ml.), the mixt. was allowed to stand 10 min. at room temp., mixed with ice, the mixt. neutralized with 30% NaOH, and the product extd. with Et_2O and C_6H_6 to give 3.02 g. of the *trans*-oxazoline (method B). The soln. of 0.01 mole amino alcohol in 10 ml. $CHCl_3$ was mixed with 0.01 mole MeC(OEt):NH.HCl in 10 ml. $CHCl_3$, the mixt. was heated 30 min. on the steam-bath, 40 ml. Et_2O was added, the NH_4Cl removed, and the soln. evapd. to dryness (method C). Thus were prepd. the following *RCH.CHR'.N:CR".O* (X), (R, R', R", method of prepn., yields in %, and m.ps. given): *cis*-Ph, Me, Ph, A, 87.6, b_1 151; *cis*-Ph, Ph, Ph, A, 88.1–89.9%, 83–4° and 102–3° (dimorphous) (from petr. ether); *trans*-Ph, Ph, Ph, B, 73.5, 83–5° (from petr. ether), 93–4° (from MeOH) (dimorphous); *cis*-Ph, CO_2Et, Me, C, 67, $b_{0.5}$ 130–1°; *trans*-Ph, CO_2Et, Me, C, 84, b_1 108–11°; *cis-p*-$O_2NC_6H_4$, CO_2Et, Me, C, 78, 132° (from EtOH); *trans-p*-$O_2NC_6H_4$, CO_2Et, Me, C, 67, 89–90° (from cyclohexane); *trans*-Ph, Ph, Me, A, 59, $b_{0.45}$ 160–1°; *cis*-C_6H_{11}, Me, Ph, A, 94, 81–3° (from ligroine); *trans*-C_6H_{11}, Me, Ph, A, 91, b_1 128°; *cis*-Me, Ph, Ph, A, 90, $b_{0.3}$ 143°. The oxazoline (0.01 mole) was dissolved in 2–4 ml. AcSH, the soln. was heated on the steam bath for the time given, the AcSH was evapd. *in vacuo*, C_6H_6 was added and again evapd. [R, R', and R" in X, time (in hrs.) and temp. of heating, yield in %, and m.p. of RCH(SAc)CHR'NHCOR" are given]: *cis*-Ph, Me, Ph, 5, 100°, 75%, 133–5° (from iso-PrOH); *trans*-Ph, Me, Ph, 4, 100°, 72%, 128–9.5° (from iso-PrOH); same, 24 hrs. at 20°, 79%; *cis*-Ph, Ph, Ph, 4, 100°, 38%, 152° (from iso-PrOH or C_6H_6); *trans*-Ph, Ph, Ph, 2, 100°, 95%, 225° (from C_6H_6); same, 12 hrs., 20°, 76%; *trans*-Ph, CO_2Et, Ph, 4, 100°, 72%, 96–8° (from iso-PrOH); same, $CHCl_3$-COSH, 2, 100°, PhCH(SCOCHCl_2)CH(CO_2Et)NHCOPh; *trans*-Ph, CO_2Et, Me, 0.5, 100°, 92%, 97–8° (from C_6H_6-petr. ether); *trans-p*-$NO_2C_6H_4$, CO_2Et, Me, 1, 100°, 73%, 139–40° (from C_6H_6-petr. ether); *trans*-Ph, Ph, Me, 4, 100°, 41%, 167–8°; *cis*-C_6H_{11}, Me, Ph, 5, 100°, 35%, 125° (from petr. ether); *cis*-Me, Ph, Ph, 5, 100°, 54%, 120–7° (from iso-PrOH). Refluxing 9 g. I with 9 ml.

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AcSH 1 hr. on the steam-bath gave 7.9 g. II, m. 136–7° (from C_6H_6). Refluxing *trans-N-benzoyl-S-acetylamino-cyclopentanethiol* (1.32 g.) in 50 ml. EtOH 6 hrs. with 3 g. Al(iso-PrO)$_3$, treating the mixt. with 200 ml. 5% tartaric acid, and extg. the product with AcOEt gave 0.77 g. *trans-N-benzoyl-2-aminocyclopentanethiol*, m. 128–8°. Heating 9.6 g. IV with 9.6 ml. AcSH 2 hrs. on the steam-bath yielded 6.85 g. VI, m. 159–60°. Heating 1.5 g. VI in a mixt. of dioxane and water 1:1 with 4.5 g. Ag_2O, 1.5 hrs. on the steam-bath, filtering the mixt., and evapg. the filtrate gave VIII, m. 120–1° (from ligroine). Heating 8.93 g. III with 9 ml. AcSH 2 hrs. on the steam-bath, distg. off the AcSH, adding petr. ether, and boiling the product with Et_2O gave sol. V, yellow needles, m. 139–40° (from C_6H_6-petr. ether), and 1.73 g. sol. *trans-N-benzoyl-S-acetyl-2-aminocyclohexanethiol*, m. 146° (from iso-PrOH). *trans-N-Acetyl-2-aminocyclohexanol* (XI), m. 124–5° (from AcOEt), was prepd. by passing 10 min. CH_2:CO into a soln. of 1.15 g. *trans*-2-aminocyclohexanol in 30 ml. H_2O, by allowing the mixt. to stand 3 hrs., by evapg. the mixt., and crystg. the residue from AcOEt. XI and BzCl gave *trans-N-acetyl-O-benzoyl-2-aminocyclohexanol*, m. 120–1° (contrary to the literature). Adding 1.2 ml. BzCl in 10 ml. Et_2O to a cooled soln. of 1 g. cyclohexenimine (XII) in 20 ml. Et_2O and 25 ml. 20% NaOH, sepg. the Et_2O layer, and evapg. the solvent gave 1.5 g. *N-benzoylcyclohexenimine* (XIII), m. 77–8° (from C_6H_6-petr. ether). Mixing 0.3 g. XIII with 0.6 ml. AcSH gave 0.34 g. *trans-N-benzoyl-S-acetyl-2-aminocyclohexanethiol*, m. 144–5° (from C_6H_6-petr. ether). Treating a soln. of 1 g. XII in 3 ml. C_6H_6 with 2.4 ml. AcSH in 3 ml. C_6H_6 and evapg. the solvent gave 0.3 g. *trans-N-acetyl-2-aminocyclohexanethiol*, m. 120–2° (from aq. EtOH). Heating a soln. of 1.8 g. *cis*-2,3-diphenylethylenimine in 3 ml. AcSH 4 hrs. on the steam-bath, distg. the excess AcSH *in vacuo*, and crystg. the residue from aq. EtOH gave 0.9 g. *threo-PhCH(SAc)CHPhNHAc*, 140–1°. **V. Synthesis and configuration of both racemic β-phenylcysteines.** Jiří Sicher, Miroslav Svoboda, and Jiří Farkaš. *Chem. Listy* 49, 1363–74; *Collection Czechoslov. Chem. Communs.* 20, 1439–51(1955) (in English).—Contrary to AcSH, $CHCl_3COSH$ adds to PhCH:C(CO$_2$Et)NHBz (I) and *p*-O$_2$NC$_6$H$_4$CH:C(CO$_2$Et)NHBz (II) and gives *threo-PhCH(SCOCHCl$_2$)CH(CO$_2$Et)NHBz* (III) and *threo-p-O$_2$NC$_6$H$_4$CH(SCOCHCl$_2$)CH(CO$_2$Et)NHBz* (IV), resp. Hydrolysis of III gave *threo-PhCH(SH)CH(CO$_2$Et)NHBz* (V), the acetylation of which yielded *threo-PhCH(SAc)CH(CO$_2$Et)NHBz* (VI). Acidic hydrolysis of V and its *erythro*-epimer, *erythro*-PhCH(SH)CH(CO$_2$Et)NHBz (VII) (see part II above) give *trans-2,5-diphenyl-4-carboxy-2-thiazoline* (VIII) and HCl salt of *threo-PhCH(SH)CH(CO$_2$H)NH$_2$* (IX). The same result was obtained by the hydrolysis of *cis*- and *trans-ethyl ester* (X, XI) of VIII. VIII was also obtained from PhCH(OEt):NH and HCl salts of IX and *erythro*-PhCH(SH)CH(CO$_2$H)NH$_2$ (XII). The action of HCl in EtOH transformed *erythro*-PhCH(SAc)CH(CO$_2$Et)NHAc (XIII) to *erythro-PhCH(SH)CH(CO$_2$Et)NH$_2$.HCl* (XIV) and to *2-methyl-4-carbethoxy-5-phenyl-2-thiazoline* (XV). Hydrolysis of XIV, XII.HCl, or XV gave IX.HCl. XIII is thus a convenient material for the prepn. of both racemic β-phenylcysteines IX and XII. Heating 13.3 g. 2-phenyl-5-*p*-nitrobenzylideneoxazolidone in 100 ml. EtOH and 5 ml. concd. H_2SO_4 2 hrs. on the steam-bath gave 12.3 g. II, m. 165–6°. Heating a mixt. of $CHCl_3COSH$ (25 ml.) with 34 g. I 5 hrs. on the steam-bath, adding Et_2O after cooling, and crystg. the white crystals from EtOH gave 24.5 g. III, m. 128–31°. Similarly II yielded 37% IV, m. 142–3°. Refluxing 2.2 g. III with 1 g. (iso-PrO)$_3$Al in 50 ml. EtOH 4 hrs., evapg. the soln. to dryness, treating the residue with 15% tartaric acid, and extg. the cryst. product with Et_2O gave 1.2 g. V, m. 125–6° (from iso-PrOH). Similarly

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JIRI SICHER

erythro-PhCH(SCOCHCl$_2$)CH(CO$_2$Et)NHBz gave VII, m. 117-19°, quantitatively. V and Ac$_2$O in pyridine gave *PhCH(SAc)CH(CO$_2$Et)NHBz*, m. 126-8° (from iso-PrOH). Heating 1.55 g. X (for the prepn. see part II) with 20 ml. EtOH satd. with HCl 20 hrs. on the steam-bath, evapg. the soln. to dryness, dissolving the residue in H$_2$O, and extg. with Et$_2$O gave 1.28 g. of an oil which was sepd. by extn. with petr. ether into insol. VIII, m. 137-8° (from C$_6$H$_6$), and 350 mg. sol. XI, m. 65-6° (from petr. ether). Treating 620 mg. X (or XI) in 1 ml. EtOH with 150 mg. NaOH in 2 ml. EtOH, acidifying the soln. with HCl and extg. with Et$_2$O gave 520 mg. VIII, erroneously formulated as PhCH:C(CO$_2$H)NCSPh by Lurje and Gacenko (*C.A.* 47, 2168*c*); *Na salt* of VIII, m. 202°, obtained by NaOH in EtOH from X and XI; *picrate* of VIII, m. 165-7° (from iso-PrOH); *HCl salt* of VIII, m. 165-7° (from iso-PrOH-Et$_2$O). Adding PhC(OEt):NH (0.33 g.) to a soln. of 0.47 g. HCl salt of IX under N, dilg. the mixt. after 24 hrs. with H$_2$O, evapg. *in vacuo*, dissolving the residue in Et$_2$O-C$_6$H$_6$ mixt. 1:1, washing the soln. with 1% HCl, evapg. the soln., and crystg. the residue from C$_6$H$_6$ gave VIII. The same compd. was obtained from XII. Heating 1.65 g. V with 35 ml. 10% HCl 5 hrs. on the steam-bath and extg. the mixt. with Et$_2$O gave 0.35 g. VIII, and from the aq. layer 0.17 g. IX.HCl, m. 198-9° (from EtOH-petr. ether). Similarly VII gave 38% VIII and IX.HCl. Heating 28.33 g. VIII 10 hrs. on the steam-bath with 700 ml. 10% HCl gave 6.5 g. VIII.HCl by ether extn. BzOH (6.9 g.), 2.1 g. recovered VIII, and by evapn. of the aq. layer 7.1 g. of IX.HCl, m. 198-9°, which was transformed by esterification and benzoylation to *threo-PhCH(SBz)CH(CO$_2$Et)NHBz*, m. 181°. Heating a suspension of 13.25 g. PhCH:C.CO.S.CPh:N in 200 ml. EtOH satd. with HCl 4 hrs. on the steam-bath, evapg. the soln. to dryness *in vacuo*, adding H$_2$O, extg. the mixt. with Et$_2$O, evapg. the ext. to dryness, and extg. the residue with petr. ether gave 400 mg. insol. VIII, 3.6 g. sol. X, m. 93-4°, and 3.7 g. XI. Refluxing 48 g. XIII with 300 ml. EtOH (satd. with HCl) 10 hrs. on the steam-bath gave 15.1 g. XIV, m. 180-1° (from iso-PrOH-Et$_2$O), and from the mother liquors by extn. with Et$_2$O, 8.82 g. XV, b$_{0.4}$ 125-30°. Heating 7.80 g. XIV with 35 ml. 10% HCl 4 hrs. on the steam-bath gave 4.05 g. of the XII.HCl, m. 90-100° solidifying and m. again at 178-80° (from H$_2$O), 184-5° (from iso-PrOH-petr. ether). Heating 2.5 g. XV with 14 ml. 10% HCl 5 hrs. on the steam-bath gave 1 g. IX.HCl, m. 204-5° (from EtOH-Et$_2$O). Similar hydrolysis of 1.55 g. XIII yielded 220 and 50 mg. of the HCl salts of IX and XII, resp. Heating 1.55 g. XIII with 20 ml. *N* NaOH 30 min. on the steam-bath under N, acidifying the soln. with HCl to pH 2, and crystg. the crystals from H$_2$O gave 0.68 g. *erythro-PhCH(SH)CH(CO$_2$H)NHAc*, m. 160°. Heating 1.55 g. XIII with 1.4 g. Ba(OH)$_2$ in 20 ml. H$_2$O 10 hrs., removing Ba with H$_2$SO$_4$, and evapg. the filtrate yielded 800 mg. *threo-PhCH(SH)CH(CO$_2$H)NHAc*, m. 146° (from H$_2$O). Esterification of the HCl salts of IX and XII with HCl in EtOH by refluxing 1 and 6 hrs., resp., gave 43% *threo*-epimer of XIV, m. 148-9° (from EtOH-petr. ether), and XIV, resp. IX.HCl was esterified and acetylated with Ac$_2$O in pyridine to give *threo-PhCH(SAc)CH(CO$_2$Et)NHAc*, m. 105.5° (from C$_6$H$_6$-petr. ether). Similar treatment of XIV gave 55.7% XIII, m. 96° (from C$_6$H$_6$-petr. ether). *threo*-PhCH(SH)CH(CO$_2$Et)NHBz and XIV gave *threo*- and (?)*thro-PhCH(SBz)CH(CO$_2$Et)NHBz*, m. 190-2° and 160-1°, resp. VI. Configuration of 1-phenyl-1,2-propanediols. Steric course of the hydrolysis of *cis*- and *trans*-1-phenyl-2-methylethylene oxide. Miroslav Sboboda and Jiri Sicher. *Chem. Listy* 49, 1375-8;

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JIRI SICHER

Collection Czechoslov. Chem. Communs. 20, 1452-5(1955)(in English).—The reaction of HCO_2H with *cis*- (I) and *trans*-PhCH:CHMe (II) as well as the acid-catalyzed fission of *cis*- (III) and *trans*-β-methylstyrene oxide (IV) give a mixt. of epimers and are not, contrary to the general opinion, sterically homogeneous. The HCO_2H oxidation of II produced a mixt. with prevailing *erythro*-*PhCH(OH)CHMeOH* (V) whereas I yielded a mixt. with prevailing *threo*-*PhCH(OH)CHMeOH* (VI). Similar results were obtained by the fission of III and IV. Pure V and VI were prepd. by the oxidation with $KMnO_4$ of I and II, resp. The higher melting β-isomer (m. 91-2°) was found to be V, the lower melting α-isomer (m. 54-5°) was VI. Adding 5.9 II to a mixt. of 31.5 g. HCO_2H and 6.65 ml. 38.5% H_2O_2, stirring the mixt. 45 min. at 40°, allowing the mixt. to stand 15 hrs. at room temp., distg. off the excess H_2O and HCO_2H *in vacuo*, drying the residue (7.7 g.) by distn. with C_6H_6, dissolving the residue in 60 ml. Et_2O, treating the soln. with 56 ml. of an ether soln. contg. 31 mg. $LiAlH_4$ per ml., heating the mixt. 1 hr. on the steam-bath, decompg. with 4.9 ml. H_2SO_4 in 90 ml. H_2O, extg. with Et_2O, and chromatographing the residue after the evapn. of the Et_2O gave by C_6H_6 elution 1.3 g. of an oil not further identified, by elution with Et_2O 0.25 g. V, m. 85-8°, and by EtOH elution 1.27 g. VI, m. 50-52°. Similar results were obtained by hydrolyzing the intermediate half-formate with NaOH. I (b_9 51-2°, n_D^{20} 1.5399) (5.9 g.) and a mixt. of 31.5 g. HCO_2H and 6.65 ml. 38.5% H_2O_2 gave in the above manner a small amt. of impure V and 2.45 g. crude VI, m. 38-42°. III was obtained from perphthalic acid and I in a 73.8% yield, b_{10} 78-80°, and IV from perphthalic acid and II in a 79.8% yield, $b_{10.1}$ 93-5°, n_D^{20} 1.5193. Refluxing a mixt. of 1 g. IV, 5 ml. H_2O, 5 ml. dioxane, and 0.1 ml. H_2SO_4 3 hrs., neutralizing it with $NaHCO_3$, evapg. *in vacuo*, extg. the residue with Et_2O, evapg. the Et_2O, and chromatographing the residue gave 950 mg. of a semicryst. residue, $b_{0.1}$ 103-5°, yielding 800 mg. of a mixt. contg. 63% V and 37% VI (according to the effect of both isomers on the dissocn. const. of phenylboric acid). Similarly 1 g. III gave 750 mg. of a mixt., $b_{0.1}$ 93-5°, contg. 20% V and 80% VI. A soln. of 5.2 g. I in 600 ml. EtOH was treated at −40° with a soln. of 10.4 g. $KMnO_4$ and 10 g. $MgSO_4.7H_2O$ in 350 ml. H_2O, the MnO_2 was removed, washed with hot EtOH, and the filtrate was evapd. *in vacuo*. The cryst. residue was dissolved in a min. amt. H_2O, extd. with Et_2O, the Et_2O was evapd. and the residue (2.5 g.) crystd. from Et_2O-petr. ether to give V. Similarly II gave 51% VI.

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Sicher, J.

Chem

Polarographic determination of rate of periodate oxidation of epimeric open-chain 1,2-glycols. P. Zuman, J. Sicher, J. Krupička, and M. Svoboda (Czech. Acad. Sci., Prague). *Nature* 178, 1407-8(1956).—The rates of fission of a series of stereoisomeric pairs of open-chain disecondary glycols by periodic acid are detd. polarographically. The specific 2nd-order rate consts. (l. mole^{-1} sec.$^{-1}$) for the isomeric pairs (given in the order *threo*, *erythro*) in $6 \times 10^{-4}M$ KIO_4 and $6 \times 10^{-4}M$ glycol aq. soln. at 25° are as follows: 1-phenylpropane-1,2-diol, 9.2, 3.7; butane-2,3-diol, 9.9, 4.8; hexane-3,4-diol, 8.8, 4.4; octane-4,5-diol, 5.5, 4.9; 1-cyclohexylpropane-1,2-diol, 5.6, 4.5; 1-phenylpropane-1,2-diol, 10.1, 4.6; 1-(*p*-methoxyphenyl)propane-1,2-diol, 9.8, 5.2; 1-phenylbutane-1,2-diol, 6.2, 4.2; 1-phenyl-3-methylbutane-1,2-diol, 11.5, 3.7; 1,2-diphenylethane-1,2-diol, 14.2, 3.3. The effect of acid and base catalysis (using HNO_3, HOAc, NaOAc, NaH_2PO_4, and Na_2HPO_4) on the rate of reaction for the two pairs is also given. The effects of mol. structure on the rate consts. are discussed.

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Author : Zuman, P.; Sicher, J.; Kruplicka, J.; Svoboda, M.
Institut. :
Title : Stereochemical Studies. VII. Periodate Oxidation of Diastereoisomeric Diols of the Type R.CH(OH).CH(OH).R'.
Orig Pub. : Collect. czechosl. chem. commun., 1958, 23, No 7, 1237-1251

Abstract : See RZhKhim, 1958, 43207.

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N-tritylserine (II), [illegible] N-tritylserine (III), 1-trityl-2-carbomethosyethylene-imine (IV), which on reaction with $NH_2OH.HCl$ gives

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Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11467.

hydroamic acid (V); the latter adds HCl with the formation of the hydrochloride of α-amino-β-chloropropiohydroxamic acid (VI); strongly basic anion exchange resins cyclize VI to I. For comparison purposes 4-benzylamine- (IX) and 4-benzhydrylaminoisooxazolidone-3 (X) were synthesized from N-benzyl-2-carbomethoxyethyleneimine (VII) and N-benzhydryl-2-carbomethoxyethyleneimine (VIII) by the same method. 1-benzylethyleneimine-2-carbohydroxamic acid (XI) is synthesized by refluxing 87.5 gms of the methyl ester of 1,2-dibromopropionic acid [sic] in 550 ml C_6H_6 for 3 hrs with 71.4 gms triethylamine and 38.2 gms benzylamine, shaking the mixture with water for 12 hrs and allowing the mixture to stand with VII [TN: meaning appears garbled] for 48 hrs, obtained by eva-

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Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11467.

porating the benzene solution with cold (5°) hydroxylamine (prepared from 49.6 gms of the hydrochloride of hydroxylamine in 250 ml CH_3OH and 24.6 gms Na in 300 ml CH_3OH) in 50 ml CH_3OH, followed by evaporation to 200 ml at 20°. XI is isolated by dilution with water and neutralization with CH_3COOH, yield 73.6%, mp 154-155° (from 99% alcohol). For proof of structure 0.3 gm XI is hydrogenated over 0.2 gm PtO_2 in 10 ml CH_3COOH and the product is refluxed for 3 hrs with 5 ml (1 : 1) HCl (acid); paper chromatography using the system phenol-water-NH_3 has established the presence of alanine (XII) and β-alanine (7 : 3) in the reaction mixture. Dry HCl gas is passed for 30 min at 0° into 20 gms I in 200 ml benzene, followed

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0.5 liter CH_3OH with 12.5 gms Na_2CO_3 in 1 liter water, allowing to stand 4 days, and acidifying with 50 ml CH_3COOH in 200 ml alcohol; the yield is 73%, mp 139-141° (decomp; from CH_3OH). Hydrogenation and hydrolysis of the latter product give serine. The hydrogenation of 0.03 mol X in 100 ml alcohol and 1 ml CH_3COOH over PtO_2 by 800 ml H_2 gives 2.15 gms of the amide of N-benzhydrylserine, mp 142-144° (from alcohol). A mixture of 0.03 mol II, 50 ml dry pyridine, and 2.5 ml mesyl chloride is kept 24 hrs at 0.4°, diluted with 200 ml water and $CHCl_3$; III is extracted in 90% yield, mp 128° (from benzene-alcohol) 0.01 mol II in 50 ml dioxane is mixed with NH_2OH (prepared from 14 gms $NH_2OH.HCl$ in 100 ml abs

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CH_3OH and 6.9 gms Na in 80 ml CH_3OH); after 3 days the reaction mixture is diluted with 100 ml water, and neutralized with CH_3COOH; further dilution results in the formation of a precipitate (33 gms) of N-tritylserinehydroxamic acid, mp 109° (CH_3OH); the product contains 1 molecule of combined CH_3OH. A mixture of 0.02 mol III, 20 ml C_6H_6, 1 ml CH_3CH, and 2.5 gms N-ethylpiperidine is refluxed 8 hrs, diluted with 15 ml $CHCl_3$, washed with water, and evaporated; the yield of IV is 80%, mp 130-131° (from benzene-cyclohexane). On standing for 3 days a mixture of 0.18 mol IV in 100 ml dioxane and 25.2 gms $NH_2OH.HCl$ and 12.4 gms Na in 150 ml CH_3OH

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CZECHOSLOVAKIA/Organic Chemistry. Naturally Occurring Substances and their Synthetic Analogs. G-3

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CZECHOSLOVAKIA/Organic Chemistry. Theoretical and General Questions on Organic Chemistry. G-1

Abs Jour: Ref Zhur-Khim., No 13, 1958, 43207.

Author : Zuman Petr, Sicher Jiri, Krupicka Josef, Svoboda Miroslav.

Inst :

Title : Stereochemical Studies. VII. Oxidation of Diastereo-isomeric Diols of RCH(OH)CH(OH)R' Type with Periodate.

Orig Pub: Chem. listy, 1957, 51, No 6, 1068-1081.

Abstract: Polarographic study (see Communication VI, RZhKhim, 1956, 78180) of the rate of oxidation of nine pairs of acyclic diols of RCH(OH)CHOHR' type with periodate at different pH (2-7.9) and diol concentration ($6 \cdot 10^{-3} - 9 \cdot 10^{-4}$ M). Investigated were ethylene glycol (I), threo- and erythro-isomers of

Card : 1/3

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

[illegible]	: [illegible]
[illegible]	: [illegible]
ASS. JOUR.	: R.Khim., No.[illegible] 1958, No. [illegible]
AUTHOR	: Svoboda, M.; Tichy, M.; Sicher, J.
INST.	: -
TITLE	: Stereochemical Studies. XI. Synthesis of cis- and trans-2-aminocyclotetradecanol and 2-aminocyclopentadecanol
ORIG. PUB.	: Collect. czechosl. chem. commun., 1958, 23, No 10, 1958-1964; Chem. listy, 1958, 52, 1951
ABSTRACT	: In order to study the relation between the configuration and chemical and physical properties, cis- and trans-2-aminocyclotetradecanol (cis- and trans-I) and cis- and trans-2-aminocyclopentadecanol (cis- and trans-II) were synthesized. Through the hydrogenation of the oxime of 2-oxycyclotetradecanone (III) over Pt (from PtO_2) in alcohol at ~ 20° and normal pressure, cis-I was obtained, yielding 21.65 g (from 100 g III), m.p.120-121° (from
[illegible]RD:	1/8

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY :
CATEGORY :

ABS. JOUR. : RZKhim., No. 23 1959, No. 82241

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT cont'd. : yield 90%, m.p. 142-142.5° (from acetone); NBD, m.p. 162-163.5° (from alcohol). By boiling (18 hours) trans-2-phenyl-4,5-dodecamethylene-Δ^2-oxazoline with concentrated HCl in alcohol, trans-I was obtained, yielding 93.5%, m.p. 105-106° (from petroleum ether); HC, m.p. 215-216° (from alcohol-ether); BD, yield 88.5%, m.p. 170-171° (from alcohol); NBD, m.p. 192-193° (from alcohol). Analogously, from trans-2-phenyl-4,5-tridecamethylene-

CARD: 3/8

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

G

COUNTRY :
CATEGORY :

ABS. JOUR. : RZKhim., No. 23 1959, No. 82241

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : cis-I, 99.5, 148-149; trans-I, 97, 129-130;
cont'd cis-II, 103, 120-121; trans-II, 95, 107-108.
The obtained methane sulfonates being heated
with CH_3COOK in alcohol (10-30 hours at 80-
90°) are transformed into 2-phenyl-4,5-poly-
methylene-Δ^2-oxazoline, the configuration of
which is opposite to the initial amino-alco-
hols [the initial amino-alcohol, polymethy-
lene, yield in %, m.p. in °C (from petroleum
ether), b.p. in °C/mm, m.p. of picrate in °C

CARD: 5/8

G-8

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY :
CATEGORY :

ABS. JOUR. : RZKhim., No. 23 1959, No. 82241

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : are given]; cis-I, trans-dodecamethylene, [illegible], 61-63, 190-200/0.2-0.3, 159-160 (from alcohol); trans-I, cis-dodecamethylene, 7[illegible], 95-[illegible], --, 160-161 (from alcohol); cis-II, trans-tridecamethylene, [illegible], 47-48, 16[illegible]-190 [illegible], 1[illegible]-1[illegible] (from CH_3OH); trans-II, cis-tridecamethylene, 91.6, 68.5-69, 19[illegible]-2[illegible]/0.[illegible], 136.5-137 (from CH_3OH). In ZD and MBD of trans-I and trans-II left standing in dioxane, saturated with gaseous HCl, acyl

CARD: 6/[illegible]

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

C

COUNTRY :
CATEGORY :

ASS. JOUR. : RZKhim., No. 23 1959, No. 82241

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT cont'd : migrates and HC of trans-2-benzoyloxy- or 2-p-nitrobenzoyloxycycloalkylamines are formed (acyl, cycloalkyl and m.p. in °C are given); benzoyl, tetradecyl, 172-173; p-nitrobenzoyl, tetradecyl, 173-175; benzoyl, pentadecyl, 166.5-167; p-nitrobenzoyl, pentadecyl, 179-181. For cis-isomers such like migration is not observed. The configuration of the products was determined according to the various ability of acyls for migration,

CARD: 7/8

G-9

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

SICHER, J.; TICHY, M.

"Stereochemical studies. XII. Absolute configuration of conhydrine and Ψ-conhydrine." (In English)

COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS, Praha, Czechoslovakia, Vol. 23, no. 11, Nov. 1958

Monthly List of EAST EUROPEAN ACCESSIONS INDEX (EEAI), LC, Vol. 8, No. 7, July 1959, Unclas.

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

[illegible] J.; [illegible]

"Stereochemical studies. [illegible]. Effect of ring size and configuration of the rate of intramolecular cyclization of cis- and trans-2-benzamidocycloalkyl methanesulfonates." (In English)

[illegible] Praha, Czechoslovakia, Vol. 23, no. 11, Nov. 1958

Monthly list of [illegible] (USAF), 10, Vol. 8, No. 7, July 1959, Unclas.

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

[illegible]

"Stereochemical studies. XIV. Effect of ring size and configuration on the rate of intramolecular cyclization of the N-thiobenzoyl derivatives of cis- and trans-2-aminocyclanols." (In English)

COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS, Praha, Czechoslovakia, Vol. 23, no. 12, Dec. 1958

Monthly list of EAST EUROPEAN ACCESSIONS (EEAI), LC, Vol. 8, No. 7, July 1959, Unclas.

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

SOCHER - J

COUNTRY : Czechoslovakia G-2
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR : Sicher, Jiri; Svoboda, Miroslav
INST. : not given
TITLE : Stereochemical Studies VIII. Synthesis of Large and Medium Ring Cis- and Trans-Aminocycl/anols.

ORIG. PUB. : Chem. listy, 1958, 52, #8, 1580-1585. Coll. ct. czechosl. chem commun., 1958, 23, #7, 1252-1259.

ABSTRACT : The reduction of 7-12, 16 and 20 membered rings of cyclic acyloin oximes over Pt (from PtO_2) gives a high yield of a mixture of cis and trans-2-aminocycl/anols (cis- and trans-ACO). Upon the reduction of the same oximes over Na in n-C_4H_9OH the basic products are aminocyclanes, while only small amounts of ACO are formed. Cis- and trans-ACO may be separated or changed into one another by means of the following procedures: A: Recrystallization of the mixture yields the less

CARD: 1/23

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY : Czechoslovakia G-2
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : soluble cis-ACO; B: Benzoylation of the residue left after recrystallization gives a mixture of derivatives of cis-and trans-ACO, enriched in trans isomer. Reaction of HCl with the above mixture causes migration of C_6H_5CO group, and leads largely to the formation of trans-O-benzoyl-ACO. Hydrolysis of the latter compound yields trans-ACO. C: Cis-ACO may be transformed into trans-ACO by reacting cis-N-benzoyl ACO with CH_3SO_2Cl, with a subsequent cyclization, in the presence of CH_3COOK, of the cis-N-

CARD: 2/23

2

COUNTRY : Czechoslovakia G-2

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY : Czechoslovakia
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :
:
:

ORIG. PUB. :

ABSTRACT : C_6H_5COCl. Trans-N-benzoyl-ACO was obtained by a partial hydrolysis of the corresponding picrate (PK) of trans-FO. Following are enumerated ACO's from which an N-benzoyl derivative was obtained and m.p., °C of benzoyl derivative:
cis-II, 124 (from alc.-petr. eth.); trans-II, 170-172 (from alc.)
cis-III, 121-122 (from aq. alc.); trans-III, 134-135 (from aq. alc.);
cis-IV, 115-116 (from aq. alc.); trans-IV, 138-139 (from benzene);
cis-V, 152-153 (from aq. alc.); trans-V, 129-130 (from benzene);
cis-VI, 141-142 (from aq. alc.); trans-VI, 119-120 (from alc.);

CARD:

7/23

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

[illegible] : [illegible]
[illegible] :

[illegible] : [illegible], No. [illegible] 1959, No. 71412

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : cis-VII, 192-193 (from aq. alc.); trans-VII, 179-180 (from alc.);
cis-VIII, 166-167 (from aq. alc.); trans-VIII, 161-162 (from aq. alc.);
cis-IX, 138-139 (from alc.); trans-IX, 142-143 (from aq. alc.);
cis-X, 107-108 (from alc.); trans-X, 102-103 (from alc.);

Interaction of CH_3COCl with trans-N-benzoyl-II (T) yields T-acetate, m.p. 138-141.5°C (from benzene).

CARD: 8/23

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

G-2

COUNTRY : Czechoslovakia
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : When ACO was reacted with p-$NO_2C_6H_4COCl$, the corresponding N-substituted ACO was obtained: Listed below are ACO's from which N-p-nitrobenzoyl derivatives were prepared, and m.p. in °C of the derivatives.

cis-II, 128-129.5 (from alc.); trans-I, 185-187 (from alc.);
cis-III, 141-142 (from alc.); trans-III, 164-165 (from alc.);
cis-IV, 130-131 (from benzene); trans-IV, 155-156 (from benzene);
cis-V, 135 (from benzene), trans-V, 137-138 (from benzene);

CARD: 9/23

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY : Czechoslovakia

CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :

TITLE :

ORIG. PUB. :

ABSTRACT : is a list of ACO from which the N-benzoyl-O-methanesulfony derivatives were prepared, yield and derivatives m.p. in °C: cis-I, 58.7, 128-129 (from ethylacetate); trans-I, 44.2, 137-138 (from $CH_3COOC_2H_5$); cis-II, 88.5, 113-114 (from benzene-petr. eth.); trans-II, 88.5, 104-105; cis-III, 88, 127-128 (from ethylacetate), trans-III, 75, 108-110 (from ethylacetate); cis-IV, 90, 120-121 (from ethylacetate-petr. eth.); trans-IV, 86, 106-107 (from ethylacetate-petr. eth.); cis-V, [illegible], 135-136 (from ethylacetate); trans-V, 43, 109-110 (from ethylacetate); cis-VI, 89,

CARD: 11/22

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

GOVT. : Czechoslovakia
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : 119-120 (from benzene-petr. eth.); trans-VI, 95, 124-125 (from benzene-petr. eth.); cis-VII, 98, 147-148 (from ethylacetate); trans-VII, 98, 150-151 (from ethylacetate); cis-VIII, 67, 137-139 (from ethylacetate); trans-VIII, 68, 126-127 (from ethylacetate); cis-IX, 95, 122-123 (from ethylacetate); trans-IX, 81, 117-119 (from ethylacetate); cis-X, 42, 114-115 (from $CH_3COC_2H_5$-petr. eth.); trans-X, 71, 100-101 (from $CH_3COC_2H_5$). FO's were obtained by boiling 0.002 moles of N-benzoyl-ACO methanesulfonates with 0.25 g of dry CH_3COOK in 150 ml of alcohol for 16 hours.

CARD: 12/23

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APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY : Czechoslovakia
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : Listed below are the oxazolines prepared by the above reaction, their b. p. in °C/mm and m.p. of their picrates in °C: cis-2-phenyl-4,5-pentamethylene-Δ^2-oxazoline (cis-XI), 115-117/0.35, 145-146 (from alcohol); trans-XI, 135/0.9, M.P. 54-55° (from petr. eth.) and 169-171 (from alc.). cis-2-phenyl-4,5-hexamethylene-Δ^2-oxazoline (cis XII), 121-125/0.3, M.P. from petr. eth. 50-51.5°, 163-165 (from alc.); trans-XII, 125/0.3, 168-169 (from alc.); cis-2-phenyl-4,5 heptamethylene-Δ^2-oxazoline (cis-XIII) 132-134/0.15, 155-156 (from alc.); trans-XIII, 110-114/0.06, 155-156 (from alc.);

CARD: 1/2

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

[illegible] : Czechoslovakia
[illegible] :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : cis-2-phenyl-octamethylene-Δ^2-oxazoline (cis-XIV), 138/0.4, 154-155 (from alc.); trans-XIV, (77% yield), 132-138/0.1, 150-151 (from alc.); cis-2-phenyl-4,5-nonamethylene-Δ^2-oxazoline (cis-XV), 130-135/0.05, m.p. 39-40° (from petr. eth.), 174-175 (from alc.); trans-XV, -, m.p. from petr. eth. 56-57°, 143-144 (from alc.); cis-2-phenyl-4,5-decamethylene-Δ^2-oxazoline (cis-XVI); -, m.p. from petr. eth. 54-55°, 163-164 (from alc.); trans-XVI, -, m.p. from petr. eth. 115-116°, 167-168 (from alc.); cis-2-phenyl-4,5 undecamethylene-Δ^2-oxazoline (cis-XVII), -,

CARD: 14/23

8

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY : Czechoslovakia
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : N-benzoyl-2-ethoxycyclooctylamine, m.p. 153-155° (from benzene) is formed at the same time as trans-XXI. Trans-N-benzoyl-V and $C_{19}H_{23}ON$(XX) are formed together with trans-XXV [illegible] XX [M.P. 140-141° (from petr. eth.)]. Substance XX consists apparently of a mixture of N-benzoyl-cyclodecenylamines and forms a cyclodecanone [semicarbazone, m.p. 201-202°], upon boiling with a 15% HCl. FO's are characterized by the melting points of known specimens obtained via condensation of ACO with benziminoethers. At the reaction conditions of method D (-15°C) trans-N-benzoyl-

CARD: 16/23

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

G-2

[illegible] : Czechoslovakia
[illegible] :

[illegible] : [illegible], No. 20 1959, No. 71417

[illegible] :
[illegible] :
[illegible] :

[illegible] :

[illegible] : cyclododecane (22% yield, m.p. fr. benzene 170°), (XXII) from cis-N-benzoyl-V. Upon boiling with an alcoholic solution of CH_3COOK XXII is transformed into cis-XIV. Cis-XIV ([illegible]% yield) is formed from trans-N-benzoyl-V. Cis-XVI (37% yield) may be obtained from cis-N-benzoyl-VII. Cis-XVI may be also made from trans-N-benzoyl-VII (85% yield). Trans-XVII (74.5% yield) is prepared from cis-N-benzoyl-VII. Cis-XVII (9[illegible].5% yield) is obtained from trans-N-benzoyl-VIII (~20°C). Cis-XVIII ([illegible].7% yield) is obtained from trans-N-benzoyl-IX. Trans-XIX (81.5% yield)

[illegible]:
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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY : Czechoslovakia
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71412

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : is obtained from cis-N-benzoyl-X. Cis-XIX (73% yield) is made from trans-N-benzoyl-X. 0.10 g of trans-N-benzoyl-V, in dioxane, saturated with HCl (3 days, ~20°) yielded 0.0[illegible] of trans-C-benzoyl-2-aminocyclodecanol chloride (Cl), (XXIII) m.p. 197-198° (from alk.-ether). Cis-N-benzoyl-V remains unchanged at the same conditions. 12.9 grams of a mixture of cis- and trans-N-benzoyl-V (1:1) yields, by analogous means, 4.85 grams of XXIII, from which by reverse migration of C_6H_5CO group were formed 3.2 grams of trans-N-benzoyl-V. By analogy with XXIII

CARD: 1/3

[illegible] trans-C-benzoyl-IX-Cl, almost quantitative,

CARD: 2/3

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

G-2

COUNTRY : Czechoslovakia
CATEGORY :

ABS. JOUR. : RZKhim., no. 20 1959, No. 71412

AUTHOR :
[illegible] :
TITLE :

ORIG. PUB. :

ABSTRACT : 182-183; trans-O-p-nitrobenzoyl-IX—Cl, almost quantitative, 183-185; trans-O-p-nitrobenzoyl-X-Cl, 100, 189-191. From 3.7 grams of cyclodecanol-2-one-oxime (XXIV-ketone), (m.p. from ethylacetate and benzene 102-103°) were obtained upon the reduction with Na in n-C_4H_9OH, together with a small amount of a mixture of cis- and trans-V, 1.89 grams of cyclodecylamine [(b.p. 102-110°/8-10 mm, chlorides m.p. 174-175° (from alc.-ether); picrates m.p. 234-236° (from alc.)], N-benzoyl derivatives, m.p. 142-144° (from iso-C_3H_7OH)].

CARD: 21/23

1α

CARD: 21/23

APPROVED FOR RELEASE: 03/14/2001

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

SICHER, J.

COUNTRY : Czechoslovakia

ABS. JOUR. : RZKhim., No. 20 1959, No. 71413

AUTHOR : Svoboda, Miroslav; Sicher, Jiri

INST. : Not given

TITLE : Stereochemical Studies II. Hoffmann's Dissociation of Quarternary 2-aminocyclanol Bases of Large and Medium-Sized Rings. Determination of Aminocyclanols*

ORIG. PUB. : Chem. listy, 1958, 52, #7, 1386-1395. Collect. czechosl. chem. commun. 1958, 23, #7, 1340-1350

ABSTRACT : Cis- and trans-2-aminocyclodecanol (cis- and trans-I), cis- and trans-2-aminocyclododecanol (cis- and trans-II), cis- and trans-2-aminocyclotridecanol (cis- and trans-III) and cis- and trans-2-aminocyclohexadecanol (cis- and trans-IV) were transformed into the corresponding amino bases, and were then subjected to Hoffman's degradation at 90-170°/12-14 mm. Trans-I was thus transformed into cis-1,2-epoxycyclodecane (cis-V), [1.71 gram yield from 4.14 g of ammonium salt, b.p. 94-96°/10 mm]. Trans-II yielded cis-1,2-epoxycyclododecane (cis-VI)

CARD: 1/7

13

*Structure.

G-2

COUNTRY : Czechoslovakia

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

ABS. JOUR. : RZKhim., No. 20 1959, No. 71413

AUTHOR :

INST. :

TITLE :

ORIG. PUB. :

ABSTRACT : [(91% yield, b.p. 130-133°/15mm)]. Trans-III gave cis-1,2-epoxycyclotridecane (cis-VII), [84% yield, b.p. 141-142°/15 mm]. Trans-IV led to the formation of cis-1,2-epoxycyclohexadecane (cis-VIII) (84% yield, b.p. 121-122°/0.4 mm). Cis-V was transformed (over acid Al_2O_3) into trans-1,2-cyclodecanediol (trans-IX), m.p. 45-50° (from petr. eth.) while cis-VI, cis-VII and cis-VIII, upon 1 hour boiling with 80% dioxane in the presence of a few drops of $HClO_4$ were also transformed into trans-1,2-cyclododecandiol,

CARD: 2/7

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

G-2

COUNTRY : Czechoslovakia
CATEGORY :

ABS. JOUR. : RZKhim., No. 20 1959, No. 71413

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : benzene-petr. eth.) and cis-XII, m.p. 113-114° (from petr. eth.). The results obtained prove that the initial AC has a cis-configuration. In addition they show that cis-1,2-substitutions of 10-and 12-membered rings transition into antiparallel configuration, is difficult. The latter is necessary for the formation of trans-three-membered rings. Trans-oxides are formed easily only from the ammonium bases of large ring AC. The structure of cis- and trans-XI (not described before) was elucidated on the basis of variations of the rates of

CARD: 5/7

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

COUNTRY : CZECHOSLOVAKIA
CATEGORY : Organic Chemistry. General and Theoretical Problems of Organic Chemistry
ABS. JOUR. : RZKhim., No.23 1959, No. 82175

AUTHOR : Svoboda, M.; Jonas, J.; Sicher, J.
INST. : -
TITLE : Stereochemical Studies. X. Effects of Configuration and Size of Ring upon Dissociation Constants of 2-aminocyclanols

ORIG. PUB. : Chem. Listy, 1958, 52, No 8, 1596-1602; Collect. Czechosl. Chem. Commun, 1958, 23,*

ABSTRACT : The values of dissociation constants of pK'a cis- and trans-2-aminocyclanols, derivatives of cyclopentane down to cyclotridecane, as well as of derivatives of cyclohexadecane and cyclosicosane were measured. Dependence of the dissociation constant of 2-aminocyclanols on the ring dimension is similar to the corresponding dependence discovered earlier (Pre-

*No 8, 1551-1558

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COUNTRY :
CATEGORY :

ABS. JOUR. : RZKhim., No.23 1959, No. 82175

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT cont'd. : presence of intramolecular hydrogen bonds, the formation of which is influenced primarily by reciprocal remoteness of HO- and NH_2- groups. The value of $\Delta pK'a$ [pK'a(cis) = pK'a (trans)] is a relative measure of this remoteness. The values of $\Delta pK'a$ show that in rings which are smaller than nine-membered ones, cis-isomer forms a hydrogen bridge easier than trans-isomer. Beginning with cyclononane the hydrogen bridge is formed easier in trans-

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COUNTRY :
CATEGORY :

ABS. JOUR. : RZKhim., No. 23 1959, No. 82175

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT cont'd. : isomer. Maximal remoteness of HO- and NH_2-groups is attained in 12-membered ring. The hydrochlorides (HC) of 2-aminocyclanols of the general formula $\overline{CHOHCH_2(CH_2)_{p-4}CH_2CHNH_2}\cdot$ $\cdot HCl$ (I) were prepared from alcohol solutions of 2-aminocyclanols by the addition of ether solution of HCl and by recrystallization from the mixture of C_2H_5OH + ether or iso-C_3H_7OH + ether (p in I, m.p. in °C cis- and trans-I,

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COUNTRY :
CATEGORY :

ABS. JOUR. : RZKhim., No.23 1959, No. 82175

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT (cont'd.) : pKa's cis- and trans-1, are given): 5, --, --, 9.11, 8.85; 6, --, --, 9.22, 9.07; 7, 177-179, 115-117, 9.30, 9.30; 8, 186-187, 126-128, 9.41, 9.37; 9, 170-171, 160-161, 9.36, 9.38; 10, 136-138, 176-177, 9.17, 9.23; 11, 190-192, 197-198, 9.11, 9.16; 12, 201-202, 228-229, 8.99, 9.10; 13, 191-192, 213-215 (from alcohol), 8.98, 9.07; 14, --, --, *8.94, 8.99; 16, 217-218, 184-185, 8.88, 8.90; 20, 173-174, 139-141, 8.85, 8.86. The following were also *8.93, 8.98; 15, --, --,

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COUNTRY :
CATEGORY :

ABS. JOUR. : RZKhim., No. 23 1959, No. 82175

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : determined: pK'a for cis- and trans-forms of
cont'd. N-methyl I (p=6, 9.47, 9.15) and N,N-dime-
thyl-I (p=6, 9.14, 9.03; p=10, 8.9, 8.89).
The values of pK'a were determined by the
titration of $4 \cdot 10^{-3}$ N solutions of*$(CH_3)_4NOH$
at 20° in an N_2 atmosphere. Through the methy-
lation of 1.15 g of trans-2-aminocyclanol
(14 hrs of boiling with 5 ml of 90% HCOOH and
5 ml of 40% CH_2O), trans-2-dimethylaminocyc-
lohexanol (II) is obtained; HC, m.p. 183-

*I in 80% aqueous methyl ether of
methylene glycol with 0.1 N solu-
tion of

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COUNTRY :
CATEGORY :

ABS. JOUR. : RZKhim., No.23 1959, No. 82175

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT cont'd. : 184.5° (from alcohol); picrate, m.p. 146-148.5°. Analogously to II, HC of cis-2-dimethylaminocyclohexanol, m.p. 182-183°, is obtained from the cis-compound. Similarly to II (40 hrs of boiling), from trans-2-aminocyclodecanol, trans-2-dimethylaminocyclodecanol, yielding 92%, b.p. 97°/1 mm, is obtained; HC, m.p. 127-129° (from alcohol-ether); picrate, m.p. 109-110 (from alcohol). Similarly, from the cis-compound, cis-2-dimethylaminocyclo-

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JIRI SICHER

5

Stereochemical studies. XI. Synthesis of *cis*- and *trans*-2-aminocyclotetradecanol and 2-aminocyclopentadecanol. Miroslav Svoboda, Miloš Tichý, and Jiří Sicher (Českoslov. akad. věd, Prague). *Chem. listy* 52, 1051–8 (1958); cf. *C.A.* 53, 1185g.—Hydrogenating a soln. of 100 g. cyclotetradecanol-2-one oxime in 1 l. EtOH over Adams catalyst gives 21.85 g. *cis*-2-aminocyclotetradecanol (I), m. 120–1° (C_6H_6, AcOEt, EtOH); HCl salt, m. 223–4° (EtOH-Et_2O). Boiling 18 hrs. a soln. of 18.0 g. *trans*-2-phenyl-4,5-dodecamethylene-Δ^2-oxazoline in 250 ml. EtOH and 300 ml. concd. HCl gives 12.2 g. *trans*-2-aminocyclotetradecanol (II), m. 105–6° (petr. ether); HCl salt, m. 215–16° (EtOH-Et_2O). Similarly is obtained *cis*-2-aminocyclopentadecanol, m. 89–90° (petr. ether, ligroine); HCl salt, m. 220–2.5° (EtOH-Et_2O). *trans*-2-Aminocyclopentadecanol, obtained in 82% yield from *trans*-2-phenyl-4,5-tridecamethylene-Δ^2-oxazoline, m. 93–3.5° (petr. ether); HCl salt, m. 192–3° (EtOH-Et_2O). Adding 16.0 g. NaOH in 250 ml. H_2O to a soln. of 18.2 g. I in 500 ml. C_6H_6 and then 14.6 g. BzCl portionwise with stirring and cooling gives 25.7 g. *cis*-2-benzamidocyclotetradecanol (III), m. 183–4° (EtOH). Similarly are obtained: 88.5% *trans*-2-benzamidocyclotetradecanol, m. 170–1° (EtOH); 90% *cis*-2-benzamidocyclopentadecanol, m. 142–2.5° (Me_2CO); 83% *trans*-2-benzamidocyclopentadecanol, m. 143–3.5° (Me_2CO-EtOH). Acylation according to the method described in *C.A.* 53, 1185g, yields *cis*-2-(*p*-nitrobenzamido)-cyclotetradecanol, m. 194.5–5.0° (EtOH), *trans*-2-(*p*-nitrobenzamido)cyclotetradecanol, m. 192–3° (EtOH), *cis*-2-(*p*-nitrobenzamido)cyclopentadecanol, m. 162.5–3.5° (EtOH), and *trans*-2-(*p*-nitrobenzamido)cyclopentadecanol, m. 181–2° (EtOH). A cooled soln. of 23.2 g. III in 400 ml. C_5H_5N kept with 16 g. $MeSO_2Cl$ at 0° overnight, the mixt. dild. with ice H_2O, the crystals filtered off, washed, and dried gives 28.6 g. *cis*-2-benzamidocyclotetradecyl methanesulfonate (IV), m. 148–9° (AcOEt). Similarly are obtained: 97% *trans*-2-benzamidocyclotetradecyl methanesulfonate, m. 129–30°; 100% *cis*-2-benzamidocyclopentadecyl methanesulfonate, m. 120–1°, and 91.5% *trans*-2-benzamidocyclopentadecyl methanesulfonate, m. 107–8°. Heating 30 hrs. a soln. of 26.0 g. crude IV and 20 g. anhyd. AcOK in 1500 ml. EtOH to 95° in a glass autoclave, distg. the EtOH, and extg. the product with Et_2O gives 19.05 g. *trans*-2-phenyl-4,5-dodecamethylene-Δ^2-oxazoline, $b_{0.2-0.1}$ 190–200°, m. 62–3° (petr. ether); picrate, m. 159–60° (EtOH). Analogously are obtained: 72% *cis*-2-phenyl-4,5-dodecamethylene-Δ^2-oxazoline, m. 95–6° (petr. ether) [picrate, m. 160–1° (EtOH)]; 95.6% *trans*-2-phenyl-4,5-tridecamethylene-Δ^2-oxazoline, $b_{0.15}$ 180–90°, m. 47–8° (petr. ether) [picrate, m. 149–50° (MeOH)], and 91.6% *cis*-2-phenyl-4,5-tridecamethylene-Δ^2-oxazoline, $b_{0.3}$ 195–202°, m. 68.5–9° (petr. ether); picrate, m. 136.5–7° (MeOH). When the *N*-acyl derivs. of the above aminocyclanols are subjected to acyl-migration (N → O) by dissolving 150 mg. of the compd. in 4 ml. dioxane satd. in the cold with HCl, the mixt. kept at room temp. overnight, dild. with 5 ml. dry Et_2O and allowed to stand overnight, the soln. contg. the cis isomers remains unchanged, whereas the trans isomers yield cryst. HCl salts of the following compds.: *trans*-2-benzoyloxycyclotetradecylamine, m. 171–3°, *trans*-2-(*p*-nitrobenzoyloxy)cyclotetradecylamine, m. 173–5°, *trans*-2-benzoyloxycyclopentadecylamine, m. 166.5–7°, and trans-2-(*p*-nitrobenzoyloxy)cyclopentadecylamine, m. 170–81°.

L. J. Urbánek

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SICHER, J.

Stereochemical studies. XVII. Dissociation constants of cyclohexanecarboxylic acids and cyclohexylamines and conformational equilibria. M. Tichý, J. Jonáš, and J. Sicher (Čsl. akad. věd, Prague). *Collection Czechoslov. Chem. Communs.* 24, 3434–41(1959); cf. *C.A.* 53, 21161*i*.—The free energy difference between the axial and equatorial form was found to be 1.6 ± 0.3 kcal. for the free, and 2.2 ± 0.3 kcal. for the ionized carboxyl group in the following acids: cyclohexanecarboxylic (I), *cis*- and *trans*-4-methylcyclohexanecarboxylic (II, III), *cis*-, and *trans*-3-*tert*-butylcyclohexanecarboxylic (IV, V), and *cis*- and *trans*-4-*tert*-butylcyclohexanecarboxylic (VI, VII). The calcns. were made on the basis of measurements of the dissocn. consts. in 80% Methyl Cellosolve using IV, V, VI, and VII as "axial and equatorial standards." Similar, though not so conclusive, data were collected from cyclohexylamines: cyclohexyl (VIII), *cis*- and *trans*-4-methylcyclohexyl (IX, X), and *cis*- and *trans*-4-*tert*-butylcyclohexyl (XI, XII). I, from CO_2 and $C_6H_{11}MgBr$, m. 31–2° (petr. ether at −50°). Hydrogenation of *p*-$MeC_6H_4CO_2H$ in AcOH (over PtO_2) at 45° and atm. pressure afforded II, b_{20} 139.0°, m. 28–9.5°. *p*-$Me_2CC_6H_4Ac$ (76 g.) added with stirring at 60° to a NaOBr soln. prepd. from 144 g. NaOH and 270 g. Br in 1200 ml. H_2O, the temp. allowed to rise to 95°, kept at 95° for 30 min., the mixt. dild. with H_2O, extd. with Et_2O, treated with SO_2, acidified with HCl, and the product filtered and crystd. gave 62 g. (80%) *p*-$Me_2CC_6H_4CO_2H$, m. 165–6° (ligroine). This (10 g.) hydrogenated over PtO_2 in AcOH gave 3.2 g. VI, m. 117.5–18° (from EtOH, C_6H_6, and AcOEt, successively); Me ester (from VI and CH_2N_2) b_9 102–3°, n_D^{20} 1.4557. Combined mother liquors from the crystn. of VI evapd. to dryness, dissolved in 60 ml. $(CH_3OH)_2$, refluxed 7 hrs. with 20 g. KOH, the mixt. dild. with 250 ml. H_2O, acidified with HCl, and the sepd. product crystd. from C_6H_6 gave 3.4 g. VII, m. 175.5–6°. Hydrogenation of 40 g. *p*-$AcNHC_6H_4Me$ in 100 ml. AcOH over 4 g. PtO_2 at 65°, distn. of the product at 105–9° at 0.3 mm., and crystn. (3 from cyclohexane, 2 from C_6H_6) gave 2.3 g. *N*-acetyl deriv. of X, m. 140.5–1°; refluxing with HCl afforded 65% X, b. 145–6°, n_D^{20} 1.4509; HCl salt m. 260.5–1.5° (EtOH–Me_2CO); Bz deriv. m. 180–0.5° (aq. EtOH). From the mother liquors, after the crystn. of X, 2.5 g. *N*-acetyl deriv. of IX, m. 98.5–9.5° (cyclohexane and C_6H_6) was obtained. Hydrolysis gave IX, b. 146–7°, n_D^{20} 1.4583; HCl salt m. 233–4° (EtOH–Me_2CO); Bz deriv. m. 130–0.5° (EtOH). VI (2.26 g.) in 30 ml. concd. H_2SO_4 and 15 ml. $CHCl_3$, treated at 40–5° during 30 min. with 2.7 g. NaN_3 with stirring, heated for 30 min. at 50°, the mixt. poured on ice, the aq. layer extd. with Et_2O, alkalized with KOH, the liberated base extd. with Et_2O, and the ext. dried ($MgSO_4$) and distd. gave 1.4 g. (73.7%) XI, b_{11} 76–8°, n_D^{20} 1.4679; HCl salt m. 280–1° (H_2O); Bz deriv. m. 157–7.5° (EtOH). Similarly VII was converted to 60.3% XII, b_7 77–8°, n_D^{20} 1.4648; HCl salt m. 311–12° (EtOH); Bz deriv. m. 180–0.5° (Me_2CO). The dissocn. consts. in 80% Me cellosolve at 20° (pK_a') for I through XII were: 7.43, 7.66, 7.42, 7.44, 7.96, 7.91, 7.43, 9.51, 9.44, 9.50, 9.24, 9.50.

M. Hudlický

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SICHER, J.; RAJSNER, M.; RUDINGER, J.; ECKSTEIN, M.; SORM, F.

Amino acids and peptides. XXVIII. Synthesis of threo- and erythro-dl-α,γ-diamino-β-hydroxybutyric acid (γ-aminothreonine and γ-amino-allothreonine). In English. Coll.Cz.Chem. 24 no.11:3719-3729 N '59. (EEAI 9:5)

1. Department of Organic Synthesis, Institut of Chemistry, Czechoslovak Academy of Science, Prague. 2. On leave of absence from the Medical Academy, Krakow, Poland (for Eckstein).
(Amino acids) (Peptides) (Allothreonine) (Amino group) (Threonine)

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

SICHER, J.; SIPOS, F.; JONAS, J.

Stereochemical studies. XVIII. Synthesis and dissociation constants of cis- and transcycloheptane 1,2-dicarboxylic acids. Coll Cz chem 26 no.1:262-275 Ja '61. (EEAI 10:9)

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Science, Prague.

(Stereochemistry) (Cycloheptanedicarboxylic acid)
(Cycloictanediscarboxylic acid.

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

SICHER, J.; SIPOS, F.; TICHY, M.

Stereochemical studies. XIX. Conformational studies on the 2-amino-4-t-butylcyclohexanols; synthesis of the four stereoisomers. Coll Cz Chem 26 no.3:847-866 Mr '61. (EEAI 10:9)

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Science, Prague.

(Aminobutylcyclohexanol)

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

MARES, F.; ROCEK, J.; SICHER, J.

The chromic acid oxidation of cycloparaffins; correlation between reactivity and thermochemical strain, and notes on reaction mechanism. Coll Cz Chem 26 no.9:2355-2369 '61.

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague.

(Chromic acid) (Cycloalkanes)

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

S/081/62/000/021/010/06[illegible]
B1[illegible]/B101

AUTHORS: [illegible], [illegible], [illegible], F., Panková, M.

TITLE: [illegible]. [illegible]. Conformational study of [illegible] cyclohexanols; an attempt at quantitative conformational analysis of the part played by adjacent groups and by solvolysis in [illegible] - difunctional derivatives of cyclohexane

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 21, 1962, 116, abstract 2[illegible]10 (collect. [illegible] Chem. Communs., v. 26, no. 9, [illegible], 24[illegible] - 24[illegible]. [illegible] summary in Russ.)

TEXT: [illegible] the transformation of the methane sulfonates [illegible] (cis- and trans-I), trans-2-[illegible] (II), trans-2-benzamido-[illegible], cis-2-benzamido-cis-4-[illegible] and cis-2-benzamido-trans-4-tert-[illegible] (V) which takes place in absolute alcohol in [illegible]

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[illegible]

[illegible] I, II and III [illegible] -oxazo- [illegible] mechanism, which requires that the [illegible] trans-axially. This condition is [illegible] is thus high (k is [illegible] also be fulfilled for the [illegible] $k = 292 \cdot 10^{-6}$ sec^{-1} at 60°C); it [illegible] for the [illegible] conformation of III, [illegible] the A-position, while III reacts [illegible] sec^{-1} at 60°C). Obviously the [illegible] the trans-A-position is achieved [illegible] with other conformations. [illegible] followed by closing of the [illegible] following arguments: 1) the fact [illegible] of cis-I, IV or V (with these [illegible] and ethoxy products are [illegible] a carbonium ion might form,

[illegible]

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2) the fact that there is a greater difference in reaction rates between III and cis-I, and between IV and V ($k \cdot 10^{-6}$ sec^{-1}; cis-I, 0.687; IV, [illegible]; V, [illegible] between III and II or trans-I, 3) the ratio of the reaction rates for I, II and III to the reaction rates for the corresponding N-p-nitro-benzoyl derivatives is 4, while the analogous relationship for cis-I is 1.4. This confirms that the same reaction mechanism is common to trans-I, II and III. The authors consider the fact that ethanolysis takes place faster in the case of V (O^E) than in the case of IV (O^S) is due to the steric stress being less reduced during the formation of the carbonium ion in the case of IV than in that of V; this is because in the case of IV the bulky $S-C_6H_5CONH$-group becomes close to H-trigonal C, while in the case of V it departs further from this form (by analogy with 2-hydroxycyclohexanone the angles between the C-H bond and the S-C-N bonds and A-C-N bonds are taken as being 15 and 105° respectively). Analysis of the reaction rate data shows that in the case of trans-I the form of the reaction is partially "twist" form, while the ratio between the amounts of I reacting in chair form and twist form is:

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[illegible]. The details of reaction [illegible] conformational equilibrium state [illegible]

[illegible] $k_{II} - k_I = 0.0292$;

[illegible] homogeneous. There is [illegible] of III, and that of II is [illegible] spectra show the conformation of [illegible] diaxial chair- [illegible] C_6H_5CO-group and CH_3-group [illegible] of K corresponds to 97% of the diequato- [illegible] and V have almost equa- [illegible] at 60°C cis-I is present in [illegible] The motive force of the C_6H_5CONH- group [illegible] of the oxazolinium ion in the [illegible] to 5.5 kcal; evidently.

[illegible]

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however, as ethanolysis of the [illegible] of 4-tert-butyl-2-methyl-cyclohexanol and certain preliminary [illegible]erations show, it has a higher value. To 0.01 moles of the [illegible]ding benzamido-4-tert-butylcyclohexanol dissolved in 15 ml dry [illegible] 0.02 mole of freshly distilled methane sulfonylchloride in [illegible]°C; the whole is held at 0°C for 6 - 12 hr and diluted with water. [illegible] crystals are filtered off, and washed in water and petroleum [illegible] The percentage yields and melting points, in °C, of the substances [illegible]ned are: II 78, 93 - 94; IV 78, 126.5 - 127 (from ethyl acetat[illegible]), 133 - 134 (from ethyl acetate); for the production of III, see [illegible] XIX, RZhKhim, 1962, abstract 12Zh7, melting point 140 - 141°C. [illegible] of IV dissolved in 100 ml absolute alcohol is heated with 0.0[illegible] $KOCOCH_3$ for 70 hrs at 95°C; the $KOSO_2CH_3$ is filtered off and [illegible] alcohol, the filtrate evaporated in vacuo, and the residue [illegible] ether and an aqueous Na_2CO_3 solution; the ether extracts are [illegible] water and dried. Of the oil separated, 1.05 g is analyzed [illegible]rephically on neutral Al_2O_3. 20 and 50 %, respectively, of 2-[illegible] and 6-benzamido-4-tert-butyl-cyclohexene-1 (VI and VII), 50 % of [illegible]-4-tert-butyl-ethoxy cyclohexane

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Stereochemical research...

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(VIII) and traces of 2-benz [illegible] -butyl-cyclohexanol acetate (IX), are washed out with C_6H_6 and [illegible] ethanolysis of V is carried out under the same conditions, [illegible] continues for 23 hrs; the oil separated amounts to 1.15 g, [illegible] ion on neutral Al_2O_3 has shown that it consists of 25 % VI, [illegible] % VIII, and 5 % IX. [Abstracted note: Complete translation.

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

SICHER, J.; ZAVADA, J.; SVOBODA, M.

Stereochemical studies. Part 22: Bromine addition to medium ring cycloölefins; a transannular reaction. Coll Cz Chem 27 no.8:1927-1938 Ag '62.

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague.

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

CZECHOSLOVAKIA

ŠIPOŠ, F; KRUPIČKA, J; TICHÝ, M; SICHER, J.

Czechoslovakia

Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Science -- Prague - (for all)

Prague, Collection of Czechoslovak Chemical Communications, No 9, 1962, pp 2079-2088

"Stereochemical Studies. XXIII. The 4-t-Butyl-2-Methylcyclohexanols -- Synthesis and Rates of Chromic Acid Oxidation."

(41)

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001550420009-5

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TICHY, M.; SIPOS, J.; SICHER, J. CSSR

Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Science, Prague (for all)

Prague, Collection of Czechoslovak Chemical Communications, No 12, 1962, pp 2907-2924

"Stereochemical Studies. XXIV. A Study of Ring Deformations in 2-Aminocyclohexanol Derivatives by Spectroscopic Intramolecular Hydrogen Bond Determination"

(3)

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CSSR

SICHER, J.; CHEREST, M.; GAULT, Y.; FELKIN, H.

no academic degrees indicated

Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Science, Prague, and C.N.R.S. Institute for Chemistry of Raw Materials (Institut des Substances Naturelles), Gif-sur-Yvette, France (for all)

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