"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

POLAND/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Ref Zhur-Khim, No 13, 1958, 43417.

Author : Eckstein Marian, Gorczyca Maria, Kocwa Aleksander, Zejc Alfred.

Inst :

Title : N-Oxides of Physiologically Active Substances. Part III. N-Oxides of Derivatives of Nicotinic Acid.

Orig Pub: Dissert. pharmac. PAN, 1957, 9, No 3, 197-204.

Abstract: With the view of producing antituberculotic preparations, syntheses were carried out of N-oxide derivatives of nicotinic acid (I N-oxide of the acid). By interaction of 0.01 mole ethyl ester of I (II) with 5 ml concentrated NH_4OH was obtained the amide of I (III), yield 90%, MP 282° (from

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Ref Zhur-Khim., No 13, 1958, 43417.

CH_3OH): III is also formed (with a yield of 70%) on reacting 0.01 mole II with 25 ml saturated alcoholic solution of NH_3. On heating II of III with $NH_2NH_2.H_2O$ there is obtained the hydrazide of I (IV), yield about 95%, MP 230° (from alcohol). On boiling IV with aldehydes in CH_3OH there are obtained (with a yield of 70-90%) the following hydrazones corresponding to the general formula RNHN=CHR' (R being herein and hereinafter the acid residue of I) [listing R', MP in° C (from alcohol)] : C_6H_5, 233-234; 2-OHC_6H_4, 270; 4-$N(CH_3)_2C_6H_4$, 251-252; furyl-2, 229; 5-nitrofuryl-2, 199-200. Reaction of 0.004 mole IV in 6 ml water with 2 ml 20% solution of $COCl_2$ in toluene, in the cold, gave N'-oxide of 2-(3'-pyridyl)-1-oxa-

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Ref Zhur-Khim., No 13, 1958, 43417.

3,4-diazolthione-5, yield 50%, MP 282° (from alcohol). On boiling of IV with equimolecular amounts of isothiocyanates in alcohol are formed, with yields of 70-90%, N'-oxides of 1-nicotinoyl-4-aryl(alkyl)-thiosemicarbazides (V) of the general formula RNHNHCSNHR' (listing R', MP in ° C): CH =CHCH , 190-191 (from 70% alcohol); C_6H_5-, 181 (from alcohol); 4-$CH_3C_6H_4$ (Va), 199 (from CH_3OH); 3-$NO_2C_6H_4$ (Vb), 219 (from alcohol); $C_6H_5CH_2$, 199 (from CH_3OH). On boiling of 0.0016 mole Va and Vb with 6 ml 2 N solution of NaOH were obtained N'-oxides of 1-(p-tolyl)- and 1-(m-nitrophenyl)2-(3'-pyridyl)-1,3,4-triazolthione-5, MP, respectively, 259.5° (from alcohol) and 280° (decomposes; from alcohol). Part II see RZhKhim, 1957, 54396.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

Country : POLAND G
Category : Organic Chemistry. Synthetic Organic Chemistry

Abs. Jour : Ref Zhur - Khim., No 5, 1959, No. 15402

Author : Eckstein, M.; Gorczycowa, M.; Kocwa, A.
Institut. :
Title : Amino-Oxides of Physiologically Active Compounds. II. Amino-Oxides of Derivatives of Isonicotinic Acid Hydrazide
Orig Pub. : Roczn. chem., 1957, 31, No 3, 847-854

Abstract : The study of the chemical and physiological properties of N-oxide of isonicotinic acid hydrazide (I, II acid) begun earlier (see report I, Ref Zhur-Khim, 1957, 54396) is continued. I combined with D-glucose (III) or L-arabinose (IV) in an alcoholic or aqueous medium forms N-oxide of isonicotinoylhydrazone of D-glucose (V) or L-arabinose. I with RNCS (VI) forms N-oxides of 1-isonicotinoyl-4-alkyl-(or aryl)-thiosemicarbazones (VII). During

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Country : G
Category :

Abs. Jour : Ref Zhur - Khim., No 5, 1959, No. 15402

Author :
Institut. :
Title :

Orig Pub. :

Abstract cont'd. : heating of I with Raney Ni (VIII), deaminization and reduction to isonicotinoylamide (IX) takes place. During analogous operation, N-oxide of II (X) gives II. During ammonolysis of saturated NH_3 in alcohol, N-oxide of ethyl ether of II (XI) is transformed into N-oxide of IX (XII). 8 mM of I and 8 mM of anhydrous II in 20 ml. of absolute CH_3OH are boiled for 6-7 hours and V is obtained, with yield of 50-60%, m.p. 153-155° (from aqueous CH_3OH).

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Country :
Category :

Abs. Jour : Ref Zhur - Khim., No 5, 1959, No. 15402

Author :
Institut. :
Title :

Orig Pub. :

Abstract cont'd. : 8 mM of I and 8 mM of II in 2 ml. of water are heated for 30 minutes at about 100°, 1 ml. of absolute alcohol is added and V is separated out, with yield of 80-85%, $[\alpha]^{22}D$ 53.62° (c 3.46; water); after 24 hours $[\alpha]^{22}D$ -5.78° (mutarotation). 8 mM of I and 8 mM of III in 16 ml. of absolute CH_3OH are boiled for three hours and V is obtained, with yield of 40-55%. Analogously to IV, from I and III in an aqueous medium V is synthesized, with yield of 68-80%,

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Country :
Category : G

Abs. Jour : Ref Zhur - Khim., No 5, 1959, No. 15402

Author :
Institut. :
Title :

Orig Pub. :

Abstract cont'd. : m.p. 167-168° (from alcohol), $[\alpha]^{17}D$ +3.67° (c 2.04; water); after 24 hours $[\alpha]^{17}D$ +7.59°. 4.4 mM of VI ($R=CH_2=CHCH_2$) are added to 4 mM of I in 20 ml. of alcohol, heated for 15 minutes at about 100°, left standing for 24 hours at about 20° and VII is obtained (alkyl $=CH_2=CHCH_2$), m.p. 212-213° (from alcohol). VII is obtained analogously (aryl and m.p. in °C. are given): C_6H_5, 227-229 (from alcohol); 4-$CH_3C_6H_4$, 243-244 (from CH_3OH). 0.5 g. of I and 10 g.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

Country : G
Category :

Abs. Jour : Ref Zhur - Khim., No 5, 1959, No. 15402

Author :
Institut. :
Title :

Orig Pub. :

Abstract cont'd. : of VIII in 60 ml. of alcohol are boiled for three hours, the filtrate and 15 ml. of washing alcohol are evaporated, the residue is dissolved in water, purified with carbon and IX is separated out by evaporation, m.p. 156-157° (from benzene). IX is also obtained analogously from isonicotinoyl hydrazide. 0.5 g. of X and 10 g. of VIII in 50 ml. of alcohol are boiled for about 3.5 hours, the filtrate is evaporated to about 10 ml. and II is separated

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Country :
Category : G

Abs. Jour : Ref Zhur - Khim., No 5, 1959, No. 15402

Author :
Institut. :
Title :

Orig Jut. :

Abstract cont'd. : out, m.p. 305-307°. 0.01 mole of XI in 40 ml. of saturated solution of NH_3 (gas) in absolute alcohol is left standing for about 15 hours at about 20°, and XII is separated out, with yield of 62-75%, m.p. 290-292° (from alcohol-benzene).-- V. Skorodumov

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

COUNTRY : Poland

CATEGORY : Organic Chemistry - Natural Compounds and Their Synthetic Analogues.

ABS. JOUR. : RZKhim., No. 19, 1959, No. 68068

AUTHOR : [illegible]; [illegible], M.; Kocwa, A.; [illegible]

[illegible]

TITLE : Search for New Medicinals in the Group of Xanthine-Derivatives. Part III. 7-Derivatives of Theophylline Containing Sulfur in the *

ORIG. PUB. : Dissert. pharmac. PAN, 1958, 10, No 4, 239-254

ABSTRACT : In the search for new medicinals which [illegible] theophylline, but are more [illegible] 7-(β-[illegible])-theophylline [illegible] to derivatives of theophylline [illegible], [illegible] (IIIa - h, wherein R is always theophylline-7-, a R' = CH_3, b R' = C_2H_5, c R' = C_3H_7, d R' = $C_4H_9CH_2$, e R' = [illegible], f R' = p-$CH_3C_6H_4$, g R' = o-$HOOCC_6H_4$, h R' = [illegible]), which can also be obtained by condensation [illegible] of II with $ClCH_2CH_2SR'$. [illegible] 7-(β-[illegible])-[illegible] (IV, V). IIIa - h [illegible] with H_2O_2, under [illegible] conditions, to the

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* [illegible]

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

JOURNAL : Poland
CATEGORY :

ABS. JOUR. : REKhim., No. 19, 1959, No. 68068

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : corresponding S-oxides RCH_2CH_2SCR' (VIIa - d) [illegible] (or from the IIIa - d) were obtained, on further [illegible] more drastic conditions, the sulfides [illegible] (VIIa - d). I and IV were [illegible] with thio- [illegible] or with phenyl-thiourea to the salts $RCH_2CH_2SC(=NH_2)$- [illegible] (VIIIa - d, wherein a R = H, X = Br; b R = H, [illegible]; c R = C_6H_5, X = Br; d R = C_6H_5, X = I); VIIIa was [illegible] by interchange reaction with $NaHCO_3$ (24 hours, [illegible]), to VIII (R' = H, X = HCO_3), MP 248° (from alcohol). [illegible] of VIIIa with a solution of $KHCO_3$ (24 hours, [illegible]) in lieu of the expected RCH_2CH_2SH there was obtained the disulfide $(RCH_2CH_2S)_2$, MP 253-254° (from dilute alcohol).

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

COUNTRY : Poland

CATEGORY :

ABS. JOUR. : AZKhim., No. 19, 1959, No. 68068

AUTHOR :

TITLE :

ORIG. PUB. :

ABSTRACT : To 5 ml of a solution of NaNH [illegible] alcohol, boiled 7-8 hours, and isolated the [illegible] (listing the substance, yield in %, M.P. in °C, and [illegible] of the methiodide): IIIa, 61, 87-88.5 (from alcohol), 1[illegible]-1[illegible] (from alcohol); b, 69.5, 105-106 (from alcohol), 117-1[illegible] (from alcohol); c, 8[illegible].5, 56-58 (from ether), 109 (from alcohol); d, 55.7, 117-119 (from alcohol), — ; e, 53, 1[illegible]-1[illegible], — ; f, 54, 119-120 (from alcohol), — ; g, 71.6, 143-144 (from alcohol), — . By interaction of I with [illegible]salicylic acid in C_2H_5OH, in the presence of C_2H_5-[illegible], there is obtained the Na-salt of IIIg, yield 81%, M.P. 20[illegible] (from alcohol), from which is isolated IIIg,

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

COUNTRY : Poland G-3
CATEGORY :

ABS. JOUR. : RZKhim., No. 19, 1959, No. 68068

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : MP 215° (from alcohol). 10 g I in 50 ml hot water, and 15 g KI are boiled for 12 hours, the solution is [illegible], and there is obtained IV, MP 197-199° (from [illegible]). [illegible] m-mole I and 15 m-mole [illegible] in 20 ml acetone are [illegible] 3-4 hours, diluted with CCl_4 and from the residue [illegible] V, MP 191-192° (from alcohol). To solution of 3 m-mole IIIa - h in 3 ml glacial CH_3COOH containing 1 drop $FeSO_4$, are added at 10-12° 0.34 ml 30% H_2O_2, allowed to [illegible] hours at about 10°, added 5 g ice, neutralized with [illegible] solution, evaporated to dryness at 30-40°, and the [illegible] are obtained [listing here, and thereafter, the [illegible], yield in %, and MP in °C (from alcohol)]: VIa,

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

COUNTRY : [illegible]
CATEGORY :

ASS. JOUR. : AZKhim., No. 19, 1959, No. [illegible]

[illegible] :

ORIG. PUB. :

[illegible] : [illegible]

[illegible]

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

G-3

COUNTRY : Poland
CATEGORY :

ABS. JOUR. : REKhim., No. 19, 1959, No. [illegible]

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : [illegible], Nr VIIIb, 90, [illegible] (from water). [illegible] VIIIc, [illegible], [illegible] (from acetone) [illegible], [illegible] (from alcohol-acetone). Part II see [illegible], [illegible], No [illegible], [illegible]. -- [illegible] Vitkovskiy.

CARD: 1/2

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

ECKSTEIN, M.

Country : Poland G-2
Category :

Abs. Jour : 45832

Author : Eckstein, M., Kocwa, A. and Pazdro, H.
Institut. : Not given
Title : Investigation of the Derivatives of 4-Hydroxycoumarin. I. Condensation Products of Naphthaldehydes and 4-Hydroxycoumarins. II. Condensa-
Orig Pub. : Roczniki Chem, 32, No 4, 789-800, 801-811 (1958)

Abstract : I. In the course of the investigation of anticoagulant derivatives of 4-hydroxycoumarin (I), the authors have investigated the condensation products of I with α- and β-naphthaldehydes (IIa, b) and with β-substituted α-naphthaldehydes, α-CHO- β-R-$C_{10}H_6$ (IIIa-d, where Ra = HO, Rb = CH_3O, Rc = C_2H_5O, and Rd = CH_3COO). When I is refluxed with 0.5 mol IIa (15 hrs) or with 0.5 mol IIb (0.5-1 hr) in alcohol) or in CH_3COOH, α-naphthyl- and β-naphthyldi-(4-hydroxycoumarinyl-

tion Products of Alkoxy- and Hydroxy-Derivatives of Benzaldehydes with 4-Hydroxycoumarin

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

Country : Poland G-2
Category :

Abs. Jour : 45832

Author :
Institut. :
Title :

Orig Pub. :

Abstract : 3)-methanes (IV, V) are obtained, yields 45.4 and 50%, mp 214-215° and 266-267°, respectively; on dehydration in a mixture of C_5H_5N and CH_3COCH (1 : 1) (30 min, about 20°) these products give about 87% yields of 3,3'-(α-naphthylidene)- and 3,3'-(β-naphthylidene)-4,4'-epoxydicoumarins, mp 395° and 316-317°. When I is refluxed briefly with IIIa in alcohol, 3-(2'-hydroxynaphthylidene-1')-2,4-diketochromane (VI) is obtained, regardless of the ratio of the components; yield 76%, mp 238°, acetate mp 225-226°.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

Country : Poland G-2
Category :

Abs. Jour : 45832

Author :
Institut. :
Title :

Orig. Pub. :

Abstract : When I and IIIa are refluxed in CH_3COOH (for 4 hrs), a compound (VII) melting at 305° (acetate derivative mp 282°) and a small amount of VI are obtained, the latter product being isolated from the mother liquor. VI appears to be an intermediate product in the formation of VII, since the latter is obtained by heating VI for 1 hr with 1 mol I in CH_3COOH. VII is also prepared by heating I with 0.5 mol IIIb or IIIc in CH_3COOH; the analogous reaction of I with IIId unexpec-

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

Country : Poland G-2
Category :

Abs. Jour : 45832

Author :
Institut. :
Title :

Orig Pub. :

Abstract : tedly yields a mixture of VI and VII. Attempts to carry out the condensation of I with VI acetate proved unsuccessful.

II. Alkoxybenzaldehydes condense with I on refluxing in alcohol (0.5-2 hrs) to give products analogous to those obtained from the condensation of I with IIa, b. The above procedure was used in the preparation of Ar-di-(4-hydroxycoumarinyl-3)-methanes (VIIIa-f, where (a) Ar-a-$CH_3OC_6H_4$, (b) Ar-2-$C_2H_5OC_6H_4$, (c) Ar-2,3-$(CH_3O)_2C_6H_3$,

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

Country : Poland G-2
Category :

Abs. Jour : 45832

Author :
Institut. :
Title :

Orig. Pub. :

Abstract : (d)Ar-2',5-$(CH_3O)_2C_6H_3$, (e) Ar-3-C_2H_5O-4-OHC_6H_3, and (f) Ar-2-CH_3COO-3-$CH_3OC_6H_4$) which are dehydrated as in the case of IV and V to the corresponding 3,3-(alkoxybenzylidene)4,6-epoxydicoumarins (IXa-f; see VIIIa-f for Ar-groups). 2-hydroxy-3-methoxybenzaldehyde (X) reacts analogously to IIIa to form with 1 mol I 3-(2'-hydroxy-3'-methoxybenzal)-2,4-diketochromane (XI), which on heating with a second mol of I is converted to XII; the latter product is also obtained

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Country : Poland G-2
Category :

Abs. Jour : 45832

Author :
Institut. :
Title :

Orig Pub. :

Abstract : by the condensation of I with 2 mols of X. For the purpose of comparison with XII and of con-

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

Country : Poland G-2
Category :

Abs. Jour : 45832

Author :
Institut. :
Title :

Orig Pub. :

Abstract : firming the structure of the latter, IXf was hydrolyzed with dil HCl to 3,3'-(2''-hydroxy-3''-methoxybenzylidene)-4,4''epoxydicoumarin, mp 294°. 3 mmols I and 3 mmols X in 7 ml alcohol are refluxed for 3-5 min; on cooling, XI is isolated, mp 197° (acetate mp 258-260°). When the refluxing is continued over a more extended period, a mixture of XI and XII is obtained. 6 mmols I and 3 mmols X in 10 ml CH_3COOH are refluxed for 4 hrs to give XII, yield 88%, mp 280-281°, acetate mp

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

Country : Poland G-2
Category :

Abs. Jour : 45852

Author :
Institut. :
Title :

Orig Pub. :

Abstract : 267-268°. 6 mmols I and 3 mmols X acetate in 6.5 ml abs alcohol are refluxed 1 min, allowed to stand 12 hrs, and refluxed for an additional 2 min; after 4-5 days VIIIf is isolated, mp 212-213° (from alc). 44 mol [sic] of VIIIf, 1 ml C_5H_5N, and 1 ml CH_3COOH are left to stand 12 hrs; IXf is obtained, mp 301-303° (from alc). All of the compounds prepared above except VIIIf and IXf were crystallized from CH_3COOH. The following mp's are given in °C: VIIIa, 213 (decomp);

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Country : Poland
Category : G-2

Abs. Jour : 45832

Author :
Institut. :
Title :

Orig Pub. :

Abstract : b, 229-230 (decomp); c, 282; d, 285; e, 218-220; IXa, 345; b, 345; c, 270; d, 266; e, 292; f, 301-303.

D. Vitkovskiy

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

SICHER, J.; RAJSNER, M.; HUDINGER, 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. (KRAI 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: 03/13/2001 CIA-RDP86-00513R000411930007-6

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S/081/62/000/021/021/069
B141/B101

AUTHORS: Eckstein, Marian, Cwynar, Józef

TITLE: Study of antithrombotic compounds from the group of 4-hydroxy cumarin derivatives. Part IV. Condensation products of 4-hydroxy cumarin with halogen derivatives of aromatic aldehydes

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 21, 1962, 167, abstract 21Zh150 (Dissert. pharmac. PAN., v. 14, no. 1, 1962, 29-39 [Eng.; summaries in Pol. and Russ.])

TEXT: To study their biological activity the compounds (IIIa-e) were synthesized by reactions of 4-hydroxy cumarin (I) with o-, m-, and p-Cl_6H_4CHO (IIa-c) as well as with 5-bromo furfural (IId) and 5-iodo furfural (IIe). An analogous reaction of I with 5-bromo salicyl aldehyde (IV) leads to the formation of a mixture of (V) with (VI). When I is condensed with 2-chloro-5-nitro-benzaldehyde (IIf) and 5-chloro-2-nitro-benzaldehyde (IIg) in CH_3OH, (IIIf, g) are obtained, while I with IIf in CH_3COOH and I with IIg in dioxan yield (VII) and (VIII), respectively. Dehydration of IIIa-c,f,g,

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yields (IXa-c,f,g); under analogous conditions IIId,e are resinified, probably owing to the instability of the furan ring. The antivitamin-K activity of IIIa-c is shown to be hardly inferior to that of dicumarol. Boiling a solution of 0.02 mole I and 0.01 mole II in 20 ml CH_3COOH for 8 hrs results in III (substance, gross formula, m.p. in °C): a, $C_{25}H_{15}ClO_6$, 206-207 (from CH_3COOH); b, $C_{25}H_{15}ClO_6$, 226-227 (from CH_3COOH); c, $C_{25}H_{15}ClO_6$, 252-254 (from CH_3COOH). The solution of 0.01 mole IId and 0.02 mole I is kept for 6-7 hrs at 50-55°C, whereby IIId, $C_{23}H_{13}BrO_7$, is obtained, yield 83.6%, m.p. 252-254°C (from CH_3COOH). Analogously IIIe, $C_{23}H_{13}IO_7$, is got from I and IIe in 50 ml CH_3OH, yield 75%, m.p. 178-180°C (decomposition; from acetone). By boiling a mixture of 0.002 mole I and 0.001 mole IIf for 2 hrs in 5 ml CH_3OH, IIIf, $C_{25}H_{14}ClNO_8$, is obtained, m.p. 154°C (from acetone; when further heated, this solidifies and melts again at 306-307°C). Analogously IIIg, $C_{25}H_{14}ClNO_8$, is obtained from I and IIg in 10 ml CH_3OH after 10 hrs, m.p. 198-199°C (decomposition; from acetone). To 2.5 g IIIa-c dissolved in 25 ml dry C_5H_5N 20 ml

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$(CH_3CO)_2O$ is added, and after 24 hrs (IXa-c) are separated (substance, gross formula, m.p. in °C): a, $C_{25}H_{13}ClO_5$, 365-366 (here and below from glacial CH_3COOH); b, $C_{25}H_{13}ClO_5$, 336-338; c, $C_{25}H_{13}ClO_5$, 346-347. Likewise (IXf) $C_{25}H_{12}ClNO_7$, is obtained from 0.25 g IIIf, m.p. 327-328°C (decomposition; from cyclohexanone); from 0.32 g IIIg, (IXg), $C_{25}H_{12}ClNO_7$, is obtained, m.p. 309-310°C (decomposition; from cyclohexanone). A mixture of 0.02 mole I and 0.01 mole IV is boiled for 6 hrs in 50 ml alcohol and yields V which is extracted with hot alcohol, $C_{16}H_9BrO_4$, m.p. 180°C (from glacial CH_3COOH), and VI, $C_{25}H_{13}BrO_6$, m.p. 313-314°C (from glacial CH_3COOH); and the O-acetyl derivative, $C_{27}H_{15}BrO_7$, m.p. 280°C (from CH_3COOH). A mixture of 0.004 mole I, 0.002 mole IIf, and 0.1 g CH_3COOK is boiled for 0.5 hr in 10 ml glacial CH_3COOH and VII is obtained, $C_{25}H_{13}NO_8$, m.p. 318-320°C (from dimethyl formamide + alcohol). The solu-

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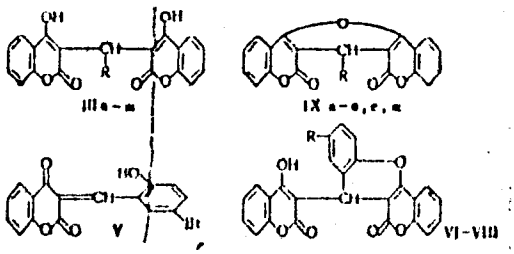
tion of 0.02 mole I and 0.01 mole IIg is boiled for 24 hrs in 50 ml dioxan and VIII is obtained, $C_{25}H_{13}ClO_6$, m.p. 313°C (from $C_6H_5NO_2$). Previous part see RZhKhim, 1959, no. 13, 45832. [Abstracter's note: Complete translation.]

Figure. Legend: (a) R = o-ClC_6H_4; (б) R = m-ClC_6H_4; (в) R = p-ClC_6H_4; (г) R = 5-bromo furyl-2; (д) R = 5-iodo furyl-2; (e) R = 2-chloro-5-nitrophenyl; (ж) R = 5-chloro-2-nitrophenyl, (VI) R = Br; (VII) R = NO_2; (VIII) R = Cl.



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ECKSTEIN, Z.

CA

11-c

Raw materials for the production of ergosterol: dried yeast and yeast extract. Z. Eckstein. *Przemysl Chem.* 4, 169-72(1948).—Dried yeast and salted and unsalted yeast ext. were investigated as a possible source of ergosterol for the manuf. of vitamin D_2. The expts. were carried out both on a lab. and plant scale, and showed that dried yeast was comparable to fresh yeast as a source of ergosterol. The output of ergosterol (in %, dry basis of input) from dried yeast ranged between 0.43% and 0.50% compared to 0.47-0.52% for fresh yeast. The output of ergosterol from yeast ext. (based on lab. expts. only) was lower, ranging from 0.2% to 0.25% for salted ext. and from 0.018% to 0.025% for unsalted ext. Frank Gonet

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ECKSTEIN, Z.

A1
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Methods of synthesis of compounds labelled with radioactive atoms. Z Eckstein (Przem. Chem., 1950, 29, 474—484).—A review.
R. Truscott

ASM SLA METALLURGICAL LITERATURE CLASSIFICATION

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FOKSTEIN, ZYGMUNT

1/2

7 Reactions of nitroparaffins. VII. Reactions of nitromethane with acetaldehyde and amines. Zygmunt Fokstein and Ladislaus Urbanski (Inst. Technol., Warsaw, Poland). *Roczniki Chem.* 26, [illegible] (English summary, [illegible]). Addition of AcH to $MeNO_2$ gave either [illegible] $NCH_2CH(OH)CH_3$ (II), depending on reaction conditions. AcH (88 g.) in 80 ml. water added with cooling to I [illegible] $MeNO_2$ and 0.1 g. K_2CO_3 [illegible] added during [illegible] reaction to keep the pH [illegible] the mixt. [illegible] hrs. at 0° gave 75% [illegible] I. [illegible] partitioned through the Na [illegible] 0.4°, 2.2 to 3 min., n_D^{20} 1.4112; chlorination [illegible] Na salt of I gave 72% crude $MeCH(OH)CH$-[illegible] n_D^{20} 1.4917. The Na salt (157 g. of I in 800 ml. [illegible]) [illegible] ml. Br in 200 ml. CCl_4 kept below 0° gave [illegible] crude $MeCH_2OH(CHBr)NO_2$ (IV), n_D^{20} 1.4965, [illegible] IV [illegible] barium and [illegible] zinc. AcH (300 g.) in 220 ml. water added to 244 [illegible] $MeNO_2$ and [illegible] $Ca(OH)_2$ with [illegible] to 30° and the mixt. stirred 2 [illegible] hrs. at room temp. in a tightly closed vessel (pH 6.5–7.0 at end of the reaction) treated with CO_2 and 6–7 l. Et_2O, and the dried Et_2O layer distd. at 0.1 mm. Hg pressure gave 80–95% (based [illegible] $MeNO_2$) crude II [illegible] AcCl (25 g. [illegible] and 15 g. II in [illegible] ml. $CHCl_3$ [illegible] until evolution of HCl ceased gave, after distr. [illegible] cryst. prod. which filtered [illegible] g. [illegible] $MeCH(Cl)CH_2NO_2$ (V) [illegible] 57.8% from ale. III [illegible] 61.3° [illegible] $NCH_2CH(OH)CH_3$, V [illegible] distd. AcH (18 ml. in 18 ml. [illegible] IV [illegible] ized (litmus) with aq. KOH [illegible] salted out with [illegible] and [illegible] cryst. from CCl_4 and $CHCl_3$ [illegible] $O_2NCH_2CH(OH)CH_3$ [illegible] avance) of the [illegible] CHV [illegible]

VA) were prepd. from II by 2 methods [illegible] and 1.5 mols. aldehyde [illegible] SO_2, the mixt. heated several hrs. [illegible] isolated by addn. of Et_2O or ale. [illegible] V and 1.5 mols. aldehyde in CH_3 [illegible] $PhSO_3H$, ale. HCl and H_2SO_4, the [illegible] throughout the course of the reaction [illegible] isolated as in [illegible] The following 2 [illegible] methyl-[illegible] (VA, R^1 = R^2 = H [illegible] yield gives [illegible] prepd. by [illegible] VI [illegible] 71.4; CH_3 (VII) 16.1 6°, 2.7, [illegible] VIII [illegible] [37.2% by [illegible] (2%), [illegible] IX [illegible]

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Zygmunt Eckstein

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p-HOC_6H_4(X), 232–4°, 15.7; *p*-$MeOC_6H_4$ (XI), 132–[illegible]°, 14.9; $PhCH_2CH_2$ (XII), 94.5–6°, 3.7; *p* - $Me_2NC_6H_4$ (XIII), 181–3°, 8.9. In the prepn. of XI-XIII unreacted aldehyde was removed with aq. $NaHSO_3$ before isolation of the product. The VA were crystd. from alc. (VII, IX, XII, XIII), from aq. (1:1, 1:3) alc. (VI, XI), or from a mixt. (3:2) of C_6H_6 and alc. (X). They are resistant to dil. alkali (except VII), but hydrolyze in dil. acids. They give pseudonitroles with HNO_2 while II with HNO_2 splits off AcH to give the nitrolic acid. The following VA ($R^1 = R^2 = H$; R^3, m.p., and % yield given) were prepd. by method (2): *p*-ClC_6H_4, 107–9°, 36.4; *o*-HOC_6H_4 (XIV), 144–6°, 21.2; *p*-$AcNHC_6H_4$, 206–7.5°, 13.2. *m-Dioxanes* (VA, $R^1 = H$; R^2, R^3, m.p., and % yield given): *p*-$O_2NC_6H_4$, *Br*, 142–4°, 32.5; *p*-$MeOC_6H_4$, *Br*, 124–6°, 9.2; *Ph*, *Br*, 98–100°, 36.5; *p*-$O_2NC_6H_4$, *Cl*, 125–7°, 55.6; *Ph*, *Cl*, 91–2.5°, 36.5. All the VA derived from 4,6-dimethyl-*m*-dioxane are white cryst. compds. except XIII, which is yellow. XIV required 16 hrs. for its prepn. and could be made by method (2) only. VA [$R^1 = R^2 = Me$, $R^3 = H$ (XV), and $R^1 = Me$, $R^2 = Pr$, $R^3 = H$ (XVI)] were prepd. Crude II (150 g.), 600 ml. dry Me_2CO, 200 g. anhyd. $CuSO_4$, and 1 ml. alc. HCl (30%) refluxed 24 hrs., the sulfate filtered off, and the Me_2CO distd. gave an oily residue which, washed with water and distd. up to 79°/5 mm., yielded a cryst. residue of 40 g. (21.1%) XV, needles with camphoric odor, m. 73.5–5.5° (from alc.). II (30 g.), 86 g. PrAc, 100 g. anhyd. $CuSO_4$, and 1 drop alc. HCl 16 hrs. at 100° gave 15 g. (28%) XVI, needles with camphoric odor m. 68–70° (from 1:1 aq. alc.). Attempts to synthesize tetrahydrooxazine or hexahydropyrimidine derivs. from II and primary amines were unsuccessful since II decompd., splitting off AcH and forming resinous polymers. Cyclohexylamine and III gave AcH and a cryst. unstable product, m. 62–4°, presumably the salt of cyclohexylamine and the hydroxamic acid [ClC(O)NHOH].

Irving D. Spencer]

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POL.

Lekstein Z. **Comparison of Methods of Synthesis of 5-Ethyl-5 (Δ1-Cyclohexenyl) Barbituric Acid.**

„Porównanie metod syntezy kwasu 5-etylo-5(Δ1-cyklohexenylo) barbiturowego". Przemysł Chemiczny. No 8, 1953, pp. 390–391.

Some methods of synthesis of 5-ethyl-5(Δ1-cyclohexenyl) barbituric acid and its Δ2-isomer are examined from the point of view of their industrial application. The condensation of ethyl-Δ1-cyclohexenylcyanoacetate with dicyandiamide gives the best yield and can be used as an industrial method. A description is given of the method of synthesis of the ester Δ2-cyclohexenylmalonate which by condensation gives 5-ethyl-5(Δ2-cyclohexenyl) barbituric acid. Investigations on mice showed that the Δ2-isomer can be considered as a specific equivalent to Δ1-isomer.

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ECKSTEIN, Z.

POLON

The comparison of methods of synthesis of 5-ethyl-5-(1-cyclohexenyl)barbituric acid. Z. Eckstein. *Przemysl Chem.* 9, 300-4(1953)(English summary). Six methods of obtaining 5-ethyl-5-(Δ¹-cyclohexenyl)barbituric acid (I) and its Δ²-isomer (II) have been investigated from the point of view of their industrial application. MeO_2CCH_2CN (189 g.), 105 g. cyclohexanone, and 10 g. $HN(CH_2CH_2OH)_2$ were mixed, let stand 24 hrs. at room temp., neutralized with 6% HCl, extd. with C_6H_6, the ext. evapd., and the residue fractionated under reduced pressure to give 270 g. 1-cyclohexenylcyanoacetate (III), b. 136-41°. To 220 g. III and 142 g. EtBr, 28 g. metallic Na in 700 ml. abs. EtOH was added dropwise during 3-4 hrs., the mixt. heated 30-60 min. to boiling on a glycerin bath, 500 ml. EtOH distd. off, 500 ml. H_2O added, the mixt. extd. with C_6H_6, the ext. distd., and the product dried over Na_2SO_4 and distd. under reduced pressure to give 200 g. Me ethyl(1-cyclohexenyl)-cyanoacetate (IV), b. 135-8°. To 4.6 g. Na in 160 ml. abs. EtOH were added 14 g. dry urea and 44 g. IV, the mixt. warmed 5 hrs. to boiling on a glycerin bath, the excess of EtOH distd. off, the mixt. warmed again for 2 hrs. at 115-22°, dissolved in 150 ml. H_2O, and extd. with 150 ml. C_6H_6, the H_2O layer was sepd., mixed with 100 ml. C_6H_6, acidified with 100 ml. concd. HCl, and the H_2O layer sepd. and neutralized with dil. NH_4OH to give 13.2 g. 5-ethyl-5-(1-cyclohexenyl)-4-iminobarbituric acid (V), white, m. 253°.

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Z. ECKSTEIN

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V (51.5 g.) was dissolved in 420 ml. dild. H_2SO_4, warmed for 45 min. and cooled to give 46.5 g. crude I, m. 171-3° (from H_2O). IV (55 g.) was poured into a soln. of 12 g. Na in 300 ml. abs. EtOH with dissolved 50 g. dry guanidine carbonate, the mixt. warmed 4.5 hrs., the ppt. filtered off, washed with abs. alc., and dissolved in dild. HCl, the soln. extd. with 100 ml. C_6H_6, and H_2O layer treated with 5% aq. NH_3 to give 42 g. 5-ethyl-5-(1-cyclohexenyl)-2,4-diimino-barbituric acid, white crystals, m. 238-40°; this dissolved in 510 ml. dild. H_2SO_4, the mixt. warmed for 60-80 min. at slow boiling and cooled, the cryst. ppt. dissolved in 10% NaOH, and the soln. acidified with HCl to give 35 g. I. To 900 ml. abs. MeOH, 35 g. Na, and 84 g. dry $H_2NC(:NH)NHCN$ (warmed until clear), 160 g. IV was added, the soln. boiled 12 hrs., the excess alc. distd. off, the concentrate cooled to 10°, 210 ml. H_2SO_4 in 510 ml. H_2O added portionwise, and the mixt. heated, boiled 12 hrs. to give 152 g. I, m. 171-3° (from H_2O). To Na in abs. EtOH was added $(CO_2Et)_2$, the mixt. kept 45 min. at 65° and cooled to 20°, a mixt. of Et α-(1-cyclohexenyl)acetate and $(CO_2Et)_2$ (each alk.-washed and dried) added dropwise during 4 hrs., the mixt. poured into dil. H_2SO_4 and ice, the H_2O layer sepd. and extd. 3 times with C_6H_6, the ext. combined with ester layer and dried ($MgSO_4$), C_6H_6 distd. off, and the product decarbonylized by distn. at 10-15 mm., and the product rectified to give di-Et α-(1-cyclohexenyl)malonate (VI), b. 137-9°. To 124 g. VI and 89 g. EtI, 12 g. Na in 300 ml. abs. EtOH was added dropwise at 60°, the mixt. heated to boiling until the reaction ended (20 min.), the excess of [illegible] distd. off to leave 86.5 g. di-Et ethyl(1-cyclohexenyl)malonate (VII), b. 147-50°. To 10.2 g. Na in 300 ml. abs. [illegible] and 58 g. dry urea, 86 g. VII was added dropwise, [illegible]

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Z. ECKSTEIN

boiled 9 hrs. on a glycerin bath, [illegible] the concentrate again heated 2 hrs. at 125-30°, and dissolved in [illegible] H_2O, the soln. extd. with CaH_2, the H_2 [illegible] and the ppt. (46.5 g. crystd from [illegible] activated C to give I [illegible] (VIII) (655 g.), b. 138 [illegible] dibromocyclohexane [illegible] g.) was treated with [illegible] di-Et ethyl(2-cyclohexenyl) [illegible] IX (135 g.) with urea 90 g. was treated [illegible] to yield 93.5 g. crude II [illegible] m. 171-4°, gave m.p. depression [illegible] g.) was treated with 131 g. dry urea [illegible] 43.5 g. Na to give 190 g. II.

Gene A. Worny

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ECKSTEIN, Z.

POL.

Reactions of nitroparaffins. VIII. Reactions of 2-bromo-2-nitropropane-1,3-diol with aldehydes. Zygmunt Eckstein (Inst. Tech., Warsaw). *Rocznik Chem.* 27, 246-52(1953)(English summary); cf. *C.A.* 49, 2437a.— Several new derivs. of 5-bromo-5-nitro-1,3-dioxane (I) were prepd. and the Br in these compds. split out by treating with $NaCH(CO_2Et)_2$, alc. KOH, or $PhCH_2NH_2$ in dioxane giving new compds. which are derivs. of 5-nitro-1,3-dioxane. 2-Bromo-2-nitropropane-1,3-diol (Ia), prepd. according to Maas [*Rec. Trav. Chim.* 16, 250(1897); 17, 384(1898)] and according to Darzens (*C.A.* 43, 128f) and contrary to the original work. The following 1,3-dioxanes were prepd. (substituents and m.ps. given): 5-Br, 5-NO_2, 2-Ph (II), 87-8.5°; 5-Br, 5-NO_2, 2-(p-$O_2NC_6H_4$) (III), 118-20°; 5-Br, 5-NO_2, 2-(p-HOC_6H_4) (IV), 111-13°; 5-Br, 5-NO_2, 2-(p-HOC_6H_4) (V), 137-9°; 5-Br, 5-NO_2, 2-(p-$MeOC_6H_4$) (VI), 87-9°; 5-Br, 5-NO_2, 2-(p-ClC_6H_4) (VII), 105-6°; 5-NO_2, 2-Ph (VIII), 126-7°; 5-NO_2, 2-(p-$O_2NC_6H_4$) (IX), 176-7°; 5-NO_2, 2-(p-$MeOC_6H_4$) (X), 143-4.5°; 5-NO_2, 2-(p-ClC_6H_4) (XI), 158-9.5°. $[C(NO_2).CH_2.O.CHPh.O.CH_2]_2$ (XII) m. 195-6°. Derivs. of I were prepd. by the following general method: 1 mole Ia, 1.5 moles of the corresponding aldehyde, and 0.1 g. $PhSO_3H$ with 200 ml. of anhyd. C_6H_6 were boiled under reflux, the water being azeotroped off with C_6H_6; Ia dissolved during the reaction; after cessation of water evolution, 1 drop H_2SO_4 was added and the mixt. was again heated to cessation of water evolution (reaction time 6-8 hrs., 12-16 hrs. in the case of o-HOC_6H_4CHO). After cooling, C_6H_6 was distd. in vacuo and the acetal was removed from residue by extn. with anhyd

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EtOH (in some cases excess aldehyde was 1st removed by shaking with satd. $NaHSO_3$), the acetal was crystd. from 96% EtOH, C_6H_6, or acetone-EtOH. IV and V are partially sol. in boiling water. Yields were 30–31%. Na (0.015 g.-atom) in 100 ml. MeOH, and 0.023 mole CH_3CO_2Et were heated 10 min. on water bath, 0.015 mole of the corresponding I added, the mixt. heated 30 min. at 50°, kept at room temp. 12–24 hrs. (after which soln. should not give an alk. reaction to phenolphthalein paper; reheated on water bath if necessary), the MeOH removed *in vacuo*, the crystals filtered off, 100 ml. water added to the filtrate, and the viscous product decanted off, washed with ether, and crystd. from 96% EtOH. The resulting products (VIII, IX, X, and XI) gave a positive pseudonitrolic test for the secondary nitro group in water-alcohol medium. When VIII was prepd. by the preceding reaction, the mother liquor after long standing gave 4.2 g. of cryst. product which did not give a test for a primary or secondary nitroparaffin, could not be purified by crystn. (m. 130–4°), and contd. 0.45–0.85% N; acetylation in quinoline with Ac_2O and soln. in 96% EtOH gave 0.5 g. of cryst. product, m. 195–6°, contd. 6.8% N, identical with XII prepd. according to Senkus (*C.A.* 45, 8032g); further concn. of mother liquor *in vacuo* gave 20.5 g. NaBr (about 80% of theory) and 23.7 g. of resinous residue from which about 10% BzH was isolated as the 2,4-dinitrophenylhydrazone. Hydrolysis of the resinous residue with alc. KOH yielded 1.8 g. of a sub-

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stance, m. 160-80°, which, by [illegible]
tone, yielded an addnl. 0.2 g. **XII**. To 9.3 g. (0.01 mole) **VI**
in 20 ml. anhyd. dioxane was added 2.2 g. (0.02 mole) of
$PhCH_2NH_2$ and the mixt. kept 2 hrs. at 70° and 6 days at
room temp. to give 1.6 g. $PhCH_2NH_2.HCl$, needles, m. 214-
16° (from acetone-EtOH); the dioxane filtrate was concd. in
vacuo to give a brown oil which, after dissolving in 10 ml
benzene, yielded 0.5 g. **X**, m. 142-4°, identical with that
obtained from **VI**. *5-Nitro-5-(hydroxymethyl)-2-phenyl-1,3-
dioxane* (**XIII**) (*C.A.* 46, 7093c) (3.0 g.) in 20 ml. EtOH and
4 ml. of 10% aq. KOH was heated 20 min. at 50°, cooled
acidified with HOAc, and diluted with water to give 1.6 g.
VIII, m. 125-7° (from EtOH), giving a pos. reaction for a
secondary NO_2 group, and a 30° m.p. depression when
mixed with **XIII**. **XIII** or **VIII** (0.1 g.) dissolved in 2 ml.
anhyd. EtOH and 1 ml. 10% aq. KOH, 1 ml. satd. aq.
$NaNO_2$ added, and the mixt. carefully acidified with dil.
H_2SO_4, gave a green-blue coloration; adding ether and
shaking yielded a green-blue org. layer. **II** (10.9 g., 0.04
mole), 9.0 g. (0.08 mole) 40% HCHO, 50 ml. EtOH, 2.5 g.
(0.05 mole) KOH, and 2 ml. water were heated at 50° to a
clear soln., left 12 hrs. (after which the pH was 7.0), and
100 ml. water added to ppt. 1.5 g. oil which crystd. on long
standing; 0.1 g. **XIII**, white flakes from 15 ml. C_6H_6, m.
125-6°, was obtained. To 4.7 g. **VIII** 2.5 g. NaOH in 20
ml. water, 2.6 ml. 33% H_2O_2 was added dropwise with the
temp. kept at 15-20°; after stirring 1 hr., a yellow ppt.
was filtered off and the filtrate was again treated with 2.6
ml. H_2O_2 as above; 0.4 g. (total) **XIII**, yellow, m. 105-6°
(from acetone), was obtained. Clayton F. Holoway

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POL.

2

3102 547.592.2-232 : 547.281.1 : 547.571 : 547.636.3-232

Eckstein Z. Aliphatic Nitrocompound Reactions. IX. Nitro-Olefine Reactions. Part I. Reactions of 1-Cyclohexenylnitromethane with Aldehydes.

„Reakcje nitrozwiązków alifatycznych. IX. Reakcje nitroolefinów. Cz. I. Reakcje 1-cykloheksenylonitrometanu z aldehydami". Roczniki Chemii (PAN). No. 1, 1954, pp. 43—54.

Crystallic 2-nitro-2-(1-cyclohexenyl)-1,3-propanediol (II) was obtained through the action of formic aldehyde on 1-cyclohexenylnitromethane (I) in a dioxane medium, in the presence of triethylamine as catalizer. Several derivatives of 5-nitro-5-(1-cyclohexenyl)1,3-dioxane were, through the action of aromatic aldehydes, obtained from compound (II). These substances are crystallic with sharp melting points. It was established that in the reaction of 1-cyclohexenylnitromethane with aromatic aldehydes, compounds are formed, which must be looked upon as derivatives of 7-nitrostilbene, because, as in the case of nitrostilbenes which are derivatives of arylnitroparaffins, they are transformed into derivatives of isoxazoline oxides and isoxazols. This reaction of 1-cyclohexenylnitromethane shows that the cyclohexenylic structure is cryptoaromatic in character. This type of transformation is as yet unknown in literature as regards certain ω-nitrostyrene derivatives.

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ECKSTEIN Z.

3

POL.

3315 517.654.5-21-1.07

Sacha A., Eckstein Z., Comparison of Methods of Synthetising 3,5-dimethyl-5-(Δ_1-Cyclohexenyl) Barbituric Acid.

„Porównanie metod syntezy kwasu 3,5-dwumetylo-5Δ_1-cykloheksenylo-barbiturowego". Przemysł Chemiczny. No. 5, 1954, pp. 263—265.

Comparative characteristics of methods of synthetising 3,5-dimethyl-5-(Δ_1-cyclohexenyl) barbituric acid are given with reference to their industrial application. It was established that methods consisting in isolating intermediate iminocompounds yielded pure 3,5-dimethyl-5-(Δ_1-cyclohexenyl) barbituric acid or products which can be readily purified.

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ECKSTEIN, Zyg.

CZYZYK, Artur; ECKSTEIN, Zygmunt

Significance of vegetable and fruits foods in the treatment of diabetes mellitus. Polski tygod. lek. 9 no.47:1525-1531 22 Nov 54.

1. Z III Kliniki Chorob Wewnetrznych; kierownik: prof. dr J.Wegierko. Z Katedry Technologii Org. II Politechniki Warszawskiej; kierownik: prof. dr T.Orhanski.

(DIABETES MELLITUS, therapy, fruit & vegetable diets)
(DIETS, in various diseases, diabetes mellitus, fruit & vegetable diets)
(VEGETABLES, ther. of diabetes mellitus with vegetable diets)
(FRUITS, ther. of diabetes mellitus with fruit diets)

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ECKSTEIN, Z.; SACHA, A.

"Comparative characteristics of Methods of Synthesizing 3, 5-dimethyl-5 -, -cyclohexnyl Barbituric Acid." P. 263. (PRZEMYSL CHEMICZNY, Vol. 10, No. 5, May 1954, Warszawa, Poland)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4, No. 1, Jan. 1955 Uncl.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

ECKSTEIN, Z.

"Institute of Organic Chemistry of the Academy of Sciences of the USSR in Moscow", p. 506, (PRZEMYSL CHEMICZNY, Vol. 10, No. 10, Oct. 1954, Warszawa, Poland)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4, No. 5, May 1955, Uncl.

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ECKSTEIN, Z.

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POL

Reaction of the aliphatic nitro compounds. IX. Reactions of nitroolefins. I. Reactions of 1-cyclohexenylnitromethane with aldehydes. Zygmunt Eckstein. Roczniki Chem. 28, 43-54(1954)(German summary). cf. C.A. 49, 2437e.—1-Cyclohexenylnitromethane (I) with HCHO gave 2-nitro-2-(1-cyclohexenyl)-1,3-propanediol (II). II with several aromatic aldehydes gave various derivs. of 5-nitro-5-(1-cyclohexenyl)-m-dioxane, cryst. products sol. in Et_2O, C_6H_6, EtOH, and Me_2CO. I and aromatic aldehydes gave derivs. of 7-nitrostilbene, confirmed by converting these products to isoxazoline oxides and isoxazoles. II was obtained by treating 54 g. I with 80.5 g. 30% HCHO in 100 ml. dioxane with 3 ml. NEt_3 (catalyst), heating to 70-5° on a water bath with stirring, letting the product stand overnight, dilg. with 600 ml. H_2O, extg. 5 times with 100 ml. Et_2O, drying with anhyd. Na_2SO_4, concg. *in vacuo*, and crystg. from C_6H_6, m. 75.5-7°. *2-Nitro-2-(1-cyclohexenyl)-1,3-diacetoxypropane*, m. 68-9° (from alc.), was obtained by heating 2 g. II in 20 ml. pyridine and 4 ml. AcOH 30 min. on a water bath, pouring the mixt. into 250 ml. H_2O with ice, and recrystg. the product (2.7 g.) from EtOH. *2-Nitro-2-(1-cyclohexenyl)-1,3-bis(p-nitrobenzoyloxy)propane*, m. 151-2°, was obtained in 2-g. yield by heating 2 g. II in 15 ml. pyridine and 2.7 g. p-$O_2NC_6H_4COCl$ 30 min. to 60° on a water bath, letting the mixt. stand overnight, dilg. with 150 ml. H_2O, and crystg. the product from EtOH. *2-Nitro-2-(1-cyclohexenyl)-1-(p-methoxyphenyl)ethylene (α-isomer)* (III), m. 97-8°, was obtained in 1.5-g. yield by treating 7.2 g. I and 6.8 g. anisaldehyde with 5 drops

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Zygomont Eckstein

[illegible] and recrystg. the product after 20 days from EtOH. *3,5-Di(1-cyclohexenyl)-4-(p-methoxyphenyl)isoxazoline oxide* was obtained by dissolving 2.6 g. III and 1.4 g. I in 60 ml. EtOH satd. with NH_3, overnight 1 g. $C_{22}H_{25}NO_3$, m. 122-4° crystd. out. *3,5-Di(1-cyclohexenyl)-4-(p-chlorophenyl)isoxazoline oxide* was obtained by dissolving 1.8 g. IV and 1.0 g. I in 20 ml. EtOH satd. with NH_3, after 20 hrs. 1.1 g. $C_{21}H_{24}ClNO_2$, m. 145-6°, crystd. out. L. M. B.

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ECKSTEIN, Z.

POL.

1. Chemotherapeutic compounds. XVIII. Hydroxamic acids. 4. Derivatives of salicylohydroxamic acid. Zygmunt Eckstein and Zdzislaw Dominiak. *Roczniki Chem.* 28, 133-43 (1954) (German summary); cf. *C.A.* 48, 13058a; 49, 6180e; Musante, *C.A.* 43, 2116b.—5,2-Br(HO)C_6H_3CONHOH (T-40) (I), 3,5,2-Br$_2$(HO)C_6H_2CONHOH (T-181) (II), and several derivs., possessing high antitubercular activity, were prepd. with high yields. I was prepd. by dissolving 270 g. NaOH and 270 g. NH_2OH HCl in H_2O at 30°, stirring 30 min., adding 1500 ml. EtOH and 693 g. 5,2-Br(HO)C_6H_3-CO_2Me heating on a water bath, cooling to 10°, acidifying with concd. HCl, washing the ppt. with 3 l. H_2O, mixing it with 1 l. EtOH, letting the mixt. stand overnight, filtering, [illegible] with 500 ml. EtOH, and drying it at 80° [illegible] I [illegible] 223-30° (decompn.). 3,5,2-[illegible] CO_2Me III [illegible] 148-30° obtained by brom-[illegible] in $CHCl_3$ with 2 moles Br at 30-40° [illegible] I prepd. Fe as catalyst and crystg. the [illegible] EtOH [illegible] with dild. NaOH to [illegible] IV [illegible] after purification). [illegible] ml. distd. [illegible] H_2O of HCl, warming [illegible] adding [illegible] g. III and 150 m. [illegible] was formed 10 min. [illegible] the mixt. 10-30 min. [illegible] filtering off the ppt. and heating it to [illegible] H_2O, cooling again and [illegible] yield 25.8 g. II m. [illegible] from C_6H_6 concd. [illegible] A.OH [illegible] obtained from the [illegible]

M. Barakan

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ECKSTEIN, Z.

ECKSTEIN, Z.

Studies in hydroxamic acids. V. Lossen's rearrangement of o-hydroxyaryl and hydroxamic acids, p. 549. (ROCZNIKI CHEMII, Warszawa, Vol. 28, no. 4, 1954.)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4, No. 6, Jan. 1955, Uncl.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

ECKSTEIN, Z.

Srodki chemiczne do walki z chwastami. Warszawa, Panstwowe Wydawn. Techniczne, 1955. 86p. (Fighting weeds by chemical means) DA Not in DLC

SOURCE: East European Accessions List (E AL) Library of Congress, Vol. 5, No. 12, December 1956.

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ECKSTEIN, Z.

4

The antituberculous properties of some derivatives of 2*H*-1,3-benzoxazine. T. Urbański, D. Górne, Z. Eckstein, and S. Słopek. *Bull. acad. polon. sci.*, Classe III, 3, 397-9 (1955).—The bacteriostatic concns. of 3,4-dihydro-3-cyclohexyl-6-bromo-2*H*-1,3-benzoxazine (m.p.: free base = 92-3°, -HCl = 240-3°) (I), 3,4-dihydro-3-benzyl-6-methyl-2*H*-1,3-benzoxazine (m.p.: free base = 79-80°, -HCl = 110-12°) (II), 3,4-dihydro-3-benzyl-6-bromo-2*H*-1,3-benzoxazine (m.p.: free base = 85-7°, -HCl = 182-4°) (III), 3,4-dihydro-3-ethyl-6-bromo-2*H*-1,3-benzoxazine (m.p.: -HCl = 171-2°) (IV), and 2,3-dihydro-2-methyl-1*H*-naphth[1,2-*e*]-*m*-oxazine (m.p.: free base = 67-8°, -HCl = 190-3°) (V) against saprophytic *Mycobacterium* were as follows: *Myc. 297*, I 3.9; II 31; III 3.9; IV 62; V 7.8; *Myc. smegmatis* I 7.8, II 31, III 7.8, IV 31, V 15.5; *Myc. H37Rv*: I 7.8, II 31, III 3.9; IV 11.5, V 15.5 (concn. units not given). John F. Lhotka

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ECKSTEIN, Z

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Reactions of 5-nitro-1,3-dioxane with diazo compounds and the synthesis of arylazonitro diols. Z. Eckstein and T. Urbański. *Bull. acad. polon. sci.*, C[redacted] 6 (1933); cf. Gochenour and Degering, *C.A.* 43, 4616f; U.S. 2,474,779 (*C.A.* 43, 8153i); U.S. 2,474,780 (*C.A.* 43, 8154a).—Derivs. of 5-nitro- (I) and 5-hydroxymethyl-*m*-dioxane (II) with diazo compds. formed azo compds. when the pH was maintained between 7.5–8.5 with KOH. II treated with base lost a mol. of CH_2O and the resulting azo compds. $R^1R^2C.O.CHR^3.C(NO_2)(N:N.Ar).CHR^4.O$ (III) were identical with those formed by I. The following III were obtained (R^1, R^2, R^3, Ar, m.p., % yield): *H, Ph, H, Ph*, 107–8.5°, 59.1; *H, Ph, H, p-ClC₆H₄, cis*-(IV), 111.5–113°, 2°, 6, and *trans*-IV, 165–7° (decompn.), 7.2; *H, Ph, H, p-O₂NC₆H₄*, 145–8° (decomp.), 27.8; *H, Ph, H, p-MeC₆H₄*, 125.5–27°, 58.2; *H, Ph, H, 2-C₁₀H₇, cis*-(V), 126.5–8.0°, 18.2, and *trans*-V, 182–3° (decompn.), 5.6; *H, Ph, Me, Ph*, 145–6°, 40.9; *H, Ph, Me, p-ClC₆H₄*, 155.5–6.5°, 66.4; *H,*

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2. Eckstein ...

Ph, Me, p-O₂N₂C₆H₄, 186-7° (decompn.), 52.4; *H, Ph, Me, p-MeC₆H₄*, 141-2°, 21.1; *H, Ph, Me, 2-C₁₀H₇*, 142-3°, 31.9; *H, p-ClC₆H₄, Me, p-O₂N₂C₆H₄*, 187-8° (decompn.), 28.0; *H, Ph, Pr, p-ONC₆H₄*, 137-8.5°, 63.4; *H, Ph, iso-Bu, p-O₂NC₆H₄*, 127.5-8.5°, 18.5; *Me, Me, Me, Ph*, 99-100.5°, 20.5; *Me, Me, Me, p-ClC₆H₄*, 138.5-40°, 38.1; *Me, Me, Me, p-O₂N₂C₆H₄, cis*-(VI), 120-1°, 11.7, *trans*-VI, 103-4°, 29.6; *Me, Me, Me, 2-C₁₀H₇*, 112-14°, 21.0; *Me, Me, H, Ph*, 94-5°, 86.8; *Me, Me, H, p-ClC₆H₄*, 142-3.5° (decompn.), 91.8; *Me, Me, H, p-O₂NC₆H₄*, 173-4° (decompn.), 72.5; *Me, Me, H, p-MeC₆H₄*, 124.5-26°, 91.2; *Me, Me, H, 2-C₁₀H₇*, 133-4°, 86.4; *Me, Me, p-O₂NC₆H₄-SC₆H₄*, 168-9° (decompn.), 73.5. The isomers of IV and V were sepd. by fractional crystn. and on hydrolysis gave the same arylazonitro diols. They also gave the same ultraviolet spectra. The following ultraviolet spectra data were obtained (max., K): *cis*-IV 291, 15,810, 405-6, 329; *trans*-IV 291-5, 13,200, 405, 261.4; *cis*-VI 286-8, 19,630, 430-5, 248.5; *trans*-VI 285, 18,010, 430-5, 233.5. Hydrolysis of III with ale. HCl gave the corresponding ketone or aldehyde, as well as $ArN{:}NC(CH_2OH)_2NO_2$ (VII). The following derivs. of VII were obtained (Ar, m.p., % yield): *Ph*, 97-9°, 82.0; *p-ClC₆H₄*, 97-8°, 72.0; *p-O₂NC₆H₄*, 114-16°, 73.6; *p-MeC₆H₄*, 95-7°, 65.6; and *2-C₁₀H₇*, 107-8°, 68.9. VII warmed with BzH and the H_2O removed azeotropically yielded cyclic acetals.

Francis Taylor, Jr.

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ECKSTEIN, Z.:

Eckstein, Z. ; Urbanski, T. On the alkylation of derivatives of 5-nitro-1, 3-dioxane. In English. p. 489.

MATEMATYKA

Vol. 3, No. 9, 1955 Warszawa, Poland

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 5, No. 10 Oct. 56

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Eckstein, Z.

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547.301.07

Eckstein Z., Nadolski J. Experiments on Pilot Plant Scale of Preparation of Allyl Alcohol.

„Próby półtechniczne otrzymywania alkoholu allilowego". Przemysł Chemiczny. No. 7, 1955, pp. 371—375, 3 figs., 4 tabs.

A description of a study of the possibility of preparing allyl alcohol from glycerol and formic acid on laboratory and pilot plant scale. The method, based on thermal decomposition (at a temperature of approx. 190 C) of glycerol formate can, after suitable modification be used to prepare allyl alcohol for synthesizing pharmaceuticals.

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ECKSTEIN, Z

Poland/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61484

Author: Eckstein, Z.

Institution: None

Title: On Problems of Production of Herbicides. I. Synthesis of 2,4-dichlorophenoxyacetic Acid (2,4-D) and of 2-methyl-4-chlorophenoxyacetic Acid (Methoxone)

Original Periodical: Z zagadnien otrzymywania srodkow chwastobojczych. I. Syntezy kwasu 2,4-dwuchlorofenoksyoctowego (2,4-D) i 2-metylo-4-chlorofenoksyoctowego (metoksonu), Przem. chem., 1955, 11, [illegible], 627-629; Polish; Russian and English resumes

Abstract: There is proposed an improved method of preparing 2,4-dichlorophenoxyacetic acid (2,4-D) (I) and 2-methyl-4-chlorophenoxyacetic acid (methoxone) (II), used as agents for control of weeds, by chlorination of phenoxyacetic acid (III) and 2-methyl-phenoxyacetic acid (IV) with a mixture of $NaClO_3$ and HCl (acid). To a

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Poland/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61484

Abstract: mixture of 0.[illegible]5 mol III in 100 ml glacial CH_3COOH and 0.6 mol HCl (d 1.17) are added dropwise 0.105 mol $NaClO_3$ in 20 ml water (temperature not above 55-60°), after addition of 1/2 of $NaClO_3$ solution the temperature is raised to 60-65°, then is held at 70° for 20 minutes, on addition of 50 ml water I is obtained, yield 87.6%, MP 136-138.5° (from water). Analogously from IV is obtained II, yield 75.2%, MP 116.5-118.5° (from water).

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ECKSTEIN, Z.

Urbanski, T.; Sobotka, W. Aliphatic nitrocompounds. XVIII. Products of nitromethane with isovaleraldehyde. p. 399.
ROCZNIKI CHEMII, Warszawa, Vol. 29, no. 2/3, 1955.

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4, no. 10, Oct. 1955, Uncl.

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ECKSTEIN, ZYGMUNT.

10

Reactions of aliphatic nitro compds. XIII. Liebermann reactions of secondary amines containing a nitro group. Tadeusz Urbański and Zygmunt Eckstein (Inst. Technol., Warsaw). *Roczniki Chem.* 29, 916-18(1955)(English summary); cf. *C.A.* 49, 11414*d*.—The Liebermann test was modified for secondary amines contg. nitro groups, which were found to interfere under the usual conditions. The soln. of nitrosamine in H_2SO_4 with phenol was not warmed. XIV. **Action of nitroparaffins on reaction between 2-aminopyridine and formaldehyde.** Tadeusz Urbański and Barbara Słowrońska-Sczaśnowa. *Ibid.* 367-74. HCHO (I), 2-aminopyridine (II), and 1-nitropropane stirred together at 20-30° and allowed to stand overnight gave *5-nitro-5-ethyl-1,3-dioxane*, m. 52-4°, and *N,N'-di(2-pyridyl)-methylenediamine* (III), m. 130-1° (methiodide, m. 225-6°), gives Liebermann test for secondary amine. III was also obtained by mixing nitro alcs. (2-ethyl-2-nitro-1,3-propanediol, 2-methyl-2-nitro-1,3-propanediol, and 2-methyl-2-nitropropanol) with II. The catalytic effect of primary and secondary nitroparaffins, $PhNO_2$, $CH_2(CO_2Et)_2$, HCl, PhOH, NH_4Cl, and 5-hydroxypyridine on the reaction of I and II to give III was also studied. XV. **Interpretation of ultraviolet absorption spectra of nitroparaffin derivatives.** Tadeusz Urbański. *Ibid.* 375-8.—On the basis of ultraviolet spectra (near 270 mμ) of some aliphatic amino nitro compds. a suggestion based on analogy with amino acids was made that the H of an amino group can be bound with both O atoms of a nitro group by means of 2 H bonds. XVI. **Products of reaction of 1-nitro-n-butane with formaldehyde and ammonia.** Tadeusz Urbański and Hanna Piotrowski. *Ibid.* 379-91.—Mixing $BuNO_2$ (I), HCHO, and NH_3 in a molar proportion 1:3:1 gave after fractional crystn. from EtOH *5-nitro-5-propyltetrahydro-1,3-oxazine-HCl* (II), m. 190-2° (dimethiodide, m. 199-200°), and *1-nitro-2-(hydroxymethyl)pentylamine-HCl* (III), m. 169-70° (O,N-di-Bz deriv., m. 101-2°). Use of 2-nitro-2-propyl-1,3-propanediol (IV) instead of I gave higher yields of II and III. I treated with 3 moles HCHO in excess NH_3 at room temp. gave *5-nitro-5-propylhexahydropyrimidine-HCl* (V), m. [illegible] (di-5-nitroso deriv., m. [illegible]). V warmed with aq. EtOH gave *2-nitro-2-propyl-1,3-propylenediamine*, m. 178-9° (di-p-nitrobenzoyl deriv., m. 216-17°). Using IV instead of I gave a better yield of V. IV warmed with excess NH_3 gave *3,7-dinitro-3,7-dipropyl-1,5-diazacyclooctane* (VI), m. 73-4° (mono-HCl salt, m. 173-4°, mono-N-nitroso deriv., m. 110-11°). VI HCl was of moderate bacteriostatic activity *in vitro* against saprophytic mycobacteria. XVII. **Products of reaction of 1-nitroisobutane with formaldehyde and ammonia.** Tadeusz Urbański and Janina Kolesińska. *Ibid.* 392-8. $Me_2CHCH_2NO_2$ (I) (1 mole) treated with 3 moles HCHO and 1 mole NH_3 gave after fractional crystn. of their HCl salts from EtOH *5-nitro-5-isopropyltetrahydroxazine* (II) HCl, m. 190° (yield

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TADEUSZ URBAŃSKI

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10%; picrate, m. 107-8° (decompn.); N-Me MeI salt, m. 201-2°], and *2-nitro-2-(hydroxymethyl)isopentylamine* (III).-HCl, m. 165-7° (yield 5%; picrate, m. 158-60°). II and III were also prepd. by treating 1 mole *2-nitro-2-isopropyl-1,3-propanediol* (m. 84-6°, obtained by heating I with HCHO at pH 7.5) with 1 mole HCHO, and 1 mole NH_3. II warmed on a steam bath with concd. HCl 6 hrs. gave HCHO and III.HCl, while III warmed with HCHO gave II. XVIII. Products of reaction of nitromethane with isovaleraldehyde. Tadeusz Urbański, Zygmunt Eckstein, and Wiesław Sabótka. *Ibid.* 399-409.—The residue from the prepn. of 1-nitro-4-methyl-2-pentanol (I) according to the method of Henry [*Rec. trav. chim.* 16, 201(1897)], Mousset [*Rec. trav. chim.* 21, 95(1902)], or Bouveault and Wahl [*Bull. soc. chim. France* 29, 643(1903)] was found to contain *5-nitro-2,8-dimethyl-4,6-nonanediol* (II), m. 92-3° (from CCl_4); diacetate, m. 108.5°. A yield of 6.5 g. II was also obtained when 29.4 g. I was treated with 20 g. isovaleraldehyde (III) in the presence of 1.6 ml. NEt_3. Chlorination and bromination of the Na salt of I led to *1-chloro-1-nitro-4-methyl-2-pentanol* (IV), b_1 98.5-9.0°, and *1-bromo-1-nitro-4-methyl-2-pentanol* (V), b_{1-2} 107-8°, resp. Treatment of IV and V with excess III led to *5-chloro-5-nitro-2,8-dimethyl-4,6-nonanediol*, m. 125-6° (from CCl_4), and *5-bromo-5-nitro-2,8-dimethyl-4,6-nonanediol* (VI), m. 133-4° (from CCl_4-$CHCl_3$), resp. II reacted with aldehydes, yielding *2-R-substituted 5-nitro-4,6-diisobutyl-1,3-dioxanes*; when R = *Ph*, *p-ClC₆H₄*, *p-MeOC₆H₄*, *p-O₂N-C₆H₄*, the m.p. was 146, 166, 148, 176°, resp. Similarly VI reacted with benzaldehyde to give *2-phenyl-5-bromo-5-nitro-4,6-diisobutyl-1,3-dioxane*, m. 153°. IV reacted with HCHO in the presence of triethylamine to give *2-chloro-2-nitro-5-methyl-1,3-hexanediol*, m. 103.5-4.0° (from CCl_4 and then C_6H_6). XIX. Preparation of alkyd resins from nitrophthalic acids and ethylene glycol. Tadeusz Urbański and Marceli Fleines. *Ibid.* 412-15.—4-Nitrophthalic acid was esterified by $(HOCH_2)_2$ at 150-8°, yielding a resin more readily than 3-nitrophthalic acid (I). Formation of a seven-membered ring by means of a hydrogen bond between the nitro group and the carboxyl group of I was suggested as an explanation.

P. Dreyfuss

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ECKSTEIN, Z

POLAND/Chemical Technology. Chemical Products and Their Application. Pesticides. H-18

Abs Jour: Referat Zhur-Khimiya, No 5, 1958, 15574.

Author : Eckstein Z., M szcynski W., Sobotka W.
Inst :
Title : On the Preparation of Herbicides. II. Syntheses of 2,4,5-Trichloro-Phenoxyacetic Acid (2,4,5-T).

Orig Pub: Przem. chem., 1956, 12, No 3, 167-169.

Abstract: Two variants (A and B) are proposed for the synthesis of 2,4,5-trichloro-phenoxyacetic acid (I), which is a herbicide and a growth regulator for some vegetables. A. 2,5-dichloraniline (II) is converted by diazotization (action of solid $NaNO_2$ on sulphate of II at 0°), and subsequent decomposition with aqueous solution of H_2SO_4 during steam-distilling, to 2,5-dichlorophenol (III) (yield 86-91%). From III, by the method described in Communication I, is prepared

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POLAND/Chemical Technology. Chemical Products and TheirApplication. Pesticides. H-18

Abs Jour: Referat Zhur-Khimiya, No 5, 1958, 15574.

2,5-dichloro-phenoxyacetic acid (IV); yield 70-75%, MP 147-148°. To 0.21 mole IV in 200 ml CH_3COOH and 35 ml concentrated HCl (acid) is added dropwise a solution of 0.07 mole $NaClO_3$ in 12 ml water (60 minutes, stirring, temperature 65-75°). The resulting technical I is washed with water and purified by preparing the Na-salt. Yield of I 83%, MP 156-157°. B. Ethyl ester of 2,4-D is nitrated with a mixture of sulfuric and nitric acid to get the ethyl ester of 5-nitro-2,4-dichloro-phenoxyacetic acid (V); yield of V 94.3%, MP 156.5-159°. On reduction of V by action of $FeSO_4$ in aqueous solution of NH_3 there is obtained 5-amino-2,4-dichloro-phenoxyacetic acid (VI); yield 84.6%, MP 169-171°. VI is converted according to Sandmeyer (diazotization of HCl-salt of VI at a temperature from -2° to 0°, and action of $CuCl_2$ and Cu) to technical I which is purified

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[illegible], [illegible]

Zygmunt Eckstein, Wieslaw Sobotka and Tadeusz Urbanski: "On Aliphatic Nitrocompounds, XX. Reactions of Nitro-Olefins. II. On Derivatives of 5-Nitro-5-(1-Cyclohexenyl)-Tetrahydro-1,3-Oxazine, "Roczniki Chemii, Vol 30, No 1, Warsaw, 1956. Published from the Research Laboratory of Organic Synthesis of the Polish Academy of Sciences in Warsaw, and the Chair of Organic Technology II, Warsaw Polytechnic, 3 Dec 54.

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Ref Zhur-Khim., No 24, 1958, 81583.

Author : Eckstein Z.
Inst :
Title : The Problem of the Preparation of Herbicides. III. The Action of Active Chlorine on Phenoxyacetic Acids.

Orig Pub: Roszn. chem., 1956, 30, No 2, 627-632.

Abstract: A method was suggested for direct chlorination (CL) of $ROCH_2COOH$ (I), where R = C_6H_5 (Ia), 2-ClC_6H_4 (Ib), 3-ClC_6H_4 (Ic), 4-ClC_6H_4 (Id), 2,4-$Cl_2C_6H_3$ (Ie), 2-$CH_3C_6H_4$ (If), or 2-CH_3-4-ClC_6H_3 (g), by the action of NaOCl (II) in glacial CH_3COOH (III) in the presence of concentrated HCl (IV). To 0.2 mole of Ia, 250 ml. of III, 0.2 grams of I_2 and 75 ml. of IV

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Abs Jour: Ref Zhur-Khim., No 24, 1958, 81583.

was added 21.6 grams of II in 40 ml of water over a period of 2 hours at 55-60°C, and after cooling 11.1 grams of I, where R 2,4,6-$Cl_3C_6H_2$ (Ih) was obtained. In the same way with a few changes the CL of the others I is carried out. The starting materials, conditions of CL, treatment after the reaction, the product of the CL, yield in %, m.p. in °C are given: 0.15 mole of Id, 200 ml. of III, 27 ml. of IV, 5.4 grams of II in 10 ml. of water, 4 hours at 45-50°C., the addition of 100 ml. of water and cooling, Ie, 61.2, 137-138; 0.15 mole of Ib, the rest as in Id, 2 hours at 45-50°C, as in Id, Ie, Id, 66.6, - ; 0.2 mole of Ie in 250 ml. of III (40 C), 0.2 grams of I , 40 ml. of IV and 14.4 grams of II in 20 ml of water, 4 hours, 45-50°C, cool-

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Ref Zhur-Khim., No 24, 1958, 81583.

ing, Ih, 47.2, 179-180 (from alcohol); 0.15 mole of Ic, 150 ml. of III, 54 ml. of IV, 0.2 grams of I_2, and 11.5 grams of II in 20 ml. of water, 4-5 hours, at 75-80°C, cooling, converting the sparingly soluble sodium salt (with 10% $NaHCO_3$), I, R = 2,4,5-$Cl_3C_6H_2$ (Ii), 9.6 grams, 157-158 (ethyl ester, m.p. 63-65°C), 0.15 moles of If, 120 ml of III (30°C), 54 ml. of IV and 11.2 grams of II in 10 ml. water, 50-55°C, I, R 4,6-Cl_2-2-$CH_3C_6H_2$ (Ij), 44.5 - ; 0.15 mole of Ig, 120 ml of III (30°C), 30 ml. of IV and 6.5 grams of II in 10 ml. of water, 50-55°C, cooling to 20°C, and Ij, 67.6, 187-188.5°C (from alcohol). Communication II, see R. Zh. Khim., 1958, 15574.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

ECKSTEIN, Zygmunt

Zygmunt Eckstein: "Some Problems of Obtaining Weed Killers. IV. Notes onnthe Synthesis of Aryloxyacetic Acid Esters." Roczniki Chemii, Vol 30, No 2, Warsaw, 1956. Published from the Chair of Organic Technology II, Warsaw Polytechnic, 2 Sep 55.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

ECKSTEIN, Z.

"Aliphatic nitro compounds. XXIII. Reactions of derivatives of 5-nitro-1, 3-dioxane. II. The product of reactions of 5-halogen-5-nitro-1, 3-dioxane with ethyl alkylmalonate."

p. 1151 (Roczniki Chemii) Vol. 30, no. 4, 1956
Warsaw, Poland

SO: Monthly Index of East European Accessions (EEAI) LC. Vol. 7, no. 4, April 1958

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

POLAND /Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour : Ref Zhur - Khim., No 10, 1958, No 32460

Author : Zygmunt Eckstein, Tadeusz Urbanski
Inst : Not given
Title : Aliphatic Nitro-Compounds. XXIV. Reaction of 5-Nitro-1,3--Dioxanes. III. Attempts to Alkylize Derivatives of 5-Nitro--1,3,-Dioxane.

Orig Pub : Roczn. chem., 1956, 30, No 4, 1163-1174

Abstract : Two stereoisomers of 5-nitro-5-(n-nitrobenzyl)-2-phenyl--1,3-dioxane (Ia, b) were obtained at the reaction of 5--nitro-2-phenyl-1,3-dioxane with n-nitrobenzylchloride (II) in CH_3ONa medium: Ia, melting point 117 to 118°, and Ib, melting point 183 to 184°. Ia was obtained also from 5--nitro-5-oxymethyl-2-phenyl-1,3-- dioxane. 5-Nitro-5-(n-nitrobenzyl)-2,2-dimethyl-1,3-dioxane (III), melting point

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POLAND / Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour : RZhKhim., No 10, 1958, No 32460

197 to 198°, was obtained similarly at thereaction of 5-nitro-2,2-dimethyl-1,3-dioxane or 5-nitro-5-oxymethyl-2,2-dimethyl-1,3-dioxane with II and CH_3ONa. 2-Nitro-2-(n-nitrobenzyl)-1,3-propandiol, melting point 86 to 87°, (IV) was obtained by the hydrolysis of Ia, Ib and III in alcohol-aqueous HCl. 2-Nitro-2-(n-nitrobenzyl)-1,3-diacetoxypropane was obtained in the result of the reaction of IV with $(CH_3CO)_2O$ in dry NC_5H_5, melting point 94 to 94.5°. 2-nitro-2-(n-nitrobenzyl)-1,3-di-(n-nitrobenzoyloxy)-propane was obtained from III at the reaction with the chloranhydride of n-nitrobenzoic acid in dry NC_5H_5, melting point 187 to 188°. A product with the melting point at 196.5 to 198° and identical to substances obtained at the reaction with III is obtained at the reaction of IV with acetone in $CHCl_3$ and in the presence of one drop of concentrated H_2SO_4. The product obtained at the reaction of

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POLAND / Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour : RZhKhim., No 10, 1958, No 32460

III with C_6H_5CHO, melting point 184 to 185°, did not show any depression of the melting temperature with Ib. 5-Nitro--5-(n-nitrobenzyl)-3-benzyltetrahydrooxazine-1,3 (V), melting point 138.5 to 139°, was obtained at the reaction of III with CH_2O and benzylamine. The same compound was obtained at the reaction of III with N-oxymethylbenzylamine. The medium of products of V hydrolysis was identified as CH_2O and chlorohydrate of 2-nitro-2-(n-nitrobenzyl)-3-(N-benzyl)-propanolamine (VI), melting point 152 to 153° (dissociates); the base, melting point 112 to 114°. See report XXIII in RZhKhim, 1958, 7958.

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ECKSTEIN, Zygmunt

POLAND / Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour : RZhKhim., No 10, 1958, No 32461

Author : Zygmunt Eckstein, Tadeusz Urbanski.
Inst : Polish Academy of Sciences Warsaw. Chair of Organic Technology, 2nd Polytechnic, Warsaw.
Title : Aliphatic Nitro-Compounds. XXV. Reactions of Derivatives of 5-Nitro-1,3-Dioxanes. IV. Products of Combination with Diazo Compounds.

Orig Pub : Roczn. Chem., 1956, 30, No 4, 1175-1187.

Abstract : As it has been established earlier (Eckstein Z., Urbanski T., Roczn. Chem., 1952, 26, 571), hydrogen at the carbon atom bonded with the nitro group of 5-nitro-1,3-dioxanes (I) is mobile and the above compounds produce reactions characteristic of secondary nitroparaffins. The combination reactions of I derivatives with dinitrated arylamines in

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POLAND / Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour : RZhKhim., No 10, 1958, No 32461

aqueous-alcohol medium in the presence of KOH confirm this observation. 23 compounds of the type R"R'COCHRC(NO_2)(N=NR'")CHRO were obtained. Identical compounds were obtained, when derivatives of 5-nitro-5-oxymethyl-1,3-dioxane were used for the combination reaction. These crystalline compounds are colored from yellow to bright red. In the cases of 5-nitro-5-(4-chlorobenzeneazo)-2-phenyl-1,3-dioxane, 5-nitro-5-(β-naphthylazo)-2-phenyl-1,3-dioxane and 5-nitro-5-(4-nitrobenzeneazo)-2,2,4,6-tetramethyl-1,3-dioxane, two isomers of each compound were obtained, the isomers differ by melting points and crystallographic structure. 5-Nitro-5-(arylazo)-2,2-dimethyl-1,3-dioxanes hydrolyzed in aqueous-alcohol HCl produce acetone and 2-nitro-2-(arylazo)-1,3-propanediol (II). These compounds are subject to conversions

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POLAND / Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour : RZhKhim., No 10, 1958, No 32461

and produce substances with higher melting points when heated in alcohol. The cyclization of II by the action of C_6H_5CHO in the presence of aminoral acid results in cyclic acetals with higher melting points. The derivatives of II are fungicides.

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ESKSTEIN Z.

POLAND/Optics - Spectroscopy K-7

Abs Jour : Ref Zhur - Fizika, No 2, 1958, No 4599

Author : Urbanski, T., Sobotka, W., Eskstein Z.
Inst : Institute of Organic Synthesis, Polish Academy of Sciences.
Title : On Ultraviolet Absorption Spectra of Some Nitro and Halogenonitrodiols.

Orig Pub : Bull. Acad. polon. sci., 1957, Cl. 3,5, No 2, 209-212

Abstract : A study was made of the influence of the length of the alkyl group (R1) and halogens (X) on ultraviolet absorption spectra of nitrodiols with the general formula R -- CHOH -- CMO_2X -- CHOH -- R. It is shown that increasing the radical R(R = CH_3, n — C_3H_7, $(CH_3)_2CHCH_2$) in the nitrodiols deepens the maximum of the band of the nitrogroup in the region of 270 -- 280 millimicrons. In chlornitrodiols these macima become weaker and in certain cases the curves have merely a point of inflection. In bromnitrodiols there are only points of inflection.

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ECKSTEIN, Z

POLAND/Organic Chemistry - Synthetic Organic Chemistry. G-2

Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11224.

Author : Eckstein, Z., Sacha, A., and Urbanski, T.
Inst : Polish Academy of Sciences
Title : On the Properties and Preparation of 1-Cycloheptenyl-Nitromethane

Orig Pub: Bull Acad Polon Sci, 1957, Cl. 3.5, No 2, 213-218, VIII (in English with a summary in Russian)

Abstract: Suberone (I) reacts in the presence of piperidine (II) with nitromethane (III) giving 1-cycloheptenyl-nitromethane (IV). The yield of IV at room temperature after 12 days is 7.2%, at 50° after 18 days - 14.2%; when I and III (1 : 3 ratio) are heated together for 24 hrs in a solution of C_6H_6 in the presence of II with the removal of the water which is formed, IV is obtained in yields of 64%, bp 84-85°/1.4-1.6 mm, $n^{20}D$ 1.4896, d^{20}_4 1.0600. The structure

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11224.

of IV is proved by the IR spectru. Curves giving the IR- and UV-spectra of IV are given.

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EDSTEIN, Z.

POLAND/Optics - Spectroscopy K-7

Abs Jour : Ref Zhur - Fizika, No 3, 1958, No 7059

Author : Sobotka W., Eskstein Z., Urbanski, T.
Inst : Not Given
Title : On Ultraviolet Absorption Spectra of Some β-Halogen Derivatives of β-Nitroalcohols. II.

Orig Pub : Bull. Acad. polon. sci., 1957, Cl. 3, 5, No 6, 653-658

Abstract : A study was made of the ultraviolet absorption spectra of 1-nitromethyl-1-oxycyclohexane (I) and its mono-chloro- and bromo-derivatives (II, III). The spectrum of I contains only a weak maximum near 270 millimicrons characteristic of the nitro group. In the case of II, the maximum of approximately 285 millimicrons is reinforced, and in the case of III the maximum of approximately 285 millimicrons occupies an intermediate place as regards intensity. For a more detailed investigation of the influence of the halogen on the alcohols with a cyclohexane ring, 2-nitro-1-cyclohexylethanol (IV) and its chloro- and bromo-derivatives (V and VI) were produced. In the spectrum of IV the maximum of the nitro group

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Eckstein, Z.

POLAND/Physical Chemistry - Molecule, Chemical Bond. B-4

Abs Jour: Referat. Zhurnal Khimiya, No 2, 1958, 3557.

Author : Z. Eckstein, T. Urbanski, W. Sobotka.
Inst : Academy of Sciences of Poland.
Title : Ultraviolet and Infrared Absorption Spectra of 2,2-Dinitropropane-1,3-Diol.

Orig Pub: Bull. Acad. polon. sci., 1957, Cl. 3,5, No 6, 679-684.

Abstract: 2,2-Dinitro-1,3-propanediol (I) in ethyl alcohol solution produces two maxima at about 276 and 363 mμ. The first maximum is characteristic of the nitrogroup. We presume, the second maximum is caused by two chromophores (NO groups) combined with two hydroxyl groups. Indeed, should the hydroxyl groups be bound producing a derivative of 1,3-dioxane, then only one maximum at about 279 mμ appears in the spectrum. The infrared spectrum of I suspended in paraffin oil does not produce the band specific of the hydroxyl group, and only a weak band at

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POLAND/Physical Chemistry - Molecule, Chemical Bond. B-4

Abs Jour: Referat. Zhurnal Khimiya, No 2, 1958, 3557.

3174 cm^{-1} appears in the CCl_4 solution, which is specific of hydroxyl groups linked with hydrogen bonds. We consider it to be possible that forms could exist, in which both the hydroxyl groups should be linked with the nitrogroup with the hydrogen bond. Three bands were found for the nitrogroup: in crystalline state 1587, 1568 and 1544 cm^{-1}, and in CCl solution - 1568, 1562 and 1527 cm^{-1}. It seems to be probable that the first band belongs to the free nitrogroup, the second - to the "semibound" nitrogroup, and the third - to the "completely bound" nitrogroup.

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ECKSTEIN, Z.

POLAND/Chemical Technology. Chemical Products and Their Application - Pesticides. H-18

Abs Jour: Referat Zhur-Khimiya, No 5, 1958, 15568.

Author : Hetnarski B., Eckstein Z., Urbanski T.
Inst :
Title : Chemical Agents for the Control of Fungi. I. Some Derivatives of S-Alkyl-, -Alkoxyalkyl- and -Arylmercury-Substituted 2-Mercapto-Benzothiazole.

Orig Pub: Przem. chem., 1957, 13, No 5, 291-293.

Abstract: By the action of alkyl-, alkoxyalkyl- and aryl-mercury halides on the Na-salt of 2-mercapto-benzothiazole (I) derivatives of I were perepared which contain the SHgR-group in 2-position, and exhibit high fungicidal activity. Listing the R, yield in %, MP in °C, concentrations inhibiting growth of Fusarium culmorum, Alternaria temuis and

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POLAND/Chemical Technology. Chemical Products and Their Application - Pesticides. H-18

Abs Jour: Referat Zhur-Khimiya, No 5, 1958, 15568.

Rhizoctonia solani: CH_3, 41.5, 114-115.5, 0.00005, 0.00001, 0.00001; C_2H_5, 41.7, 89.5-90.5, 0.00005, 0.00001, 0.00005; n-C_3H_7, 48.8, 81-81.5, 0.00005, 0.00005, 0.00005; n-C_4H_9, 75.0, 67.5-68.5, 0.00005, 0.00005, 0.00005; n-C_5H_{11}, 44.4, 58-59, 0.0005; 0.0005, 0.0005; $CH_3OC_2H_4$, 62.3, 35-37, 0.0005, 0.0005, 0.0005; p-ClC_6H_4, 64.2, 177-178.5, 0.5, 0.5, 0.5; p-$CH_3C_6H_4$, 77.5, 156-158, 0.005, 0.0005, 0.001.

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G-2

POLAND / Organic Chemistry. Synthetic Organic Chemistry.

Abs Jour : RZhKhim, No 10, 1958, No 32402

Author : Z. Eckstein, W. Ciopionko, E. Grochowski, W. Sobotka, B. Zaszczynska.

Inst : Not given

Title : To The Question of Herbicide Synthesis. V. Study of Condensation Rate of Sodium Phenolates and Chlorophenolates with Sodium Chloroacetate in Aqueous Medium.

Orig Pub : Przem. chem., 1957, 13, No 7, 390-393

Abstract : The reaction $ClCH_2COONa + NaOC{=}CRCH{=}CR'CR''{=}CH \rightarrow CH{=}CR''CR'{=}CHCR{=}COCH_2COONa$ carried out in the aqueous medium was studied on examples with (the R-s, R'-s and R''-s are enumerated): H, H, H; CH_3, H, H; Cl, Cl, H; Cl, H, Cl; Cl, Cl, Cl; and CH_3, Cl, H. The reaction was checked by the determination of the Cl^- content in the mixture. It

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Eckstein Z

POLAND / Chemical Technology. Chemical Products and Their Application. Pesticides. H

Abs Jour: Ref Zhur-Khimiya, No 9, 1959, 32537.

Author : Byrdy, S., Eckstein, Z., Sobotka, W., Winsztal, H.
Inst : Not given.
Title : Concerning the Insecticide Activity of the Conversion Products of 1,1,1,-trichlorine-2,2-bis (p-fluorphenyl)- ethane.

Orig Pub: Przem. chem., 1957, 13, No 9, 540-542.

Abstract: No abstract.

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ECKSTEIN, Z.

J. Alkiewicz, Z. ECKSTEIN, H. Halweg, P. Krakowka, T. Urbanski: "Fungistatic Actitity of Some Hydroxamic Acids," Nature, Vol. 180, No. 4596, (London) 30 November 1957, pp. 1204-1205. Published from the Department of Dermatology, Municipal Hospital No. 1, Poznan; Department of Chemistry, Institute of Technology, Warsaw; and, Laboratory of Mycology, Institute of Tuberculosis, Warsaw.

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ECKSTEIN, Z.; KRACZKIEWICZ, T.; SACHA, A.; URBANSKI, T.

On absorption spectra of 2-nitro-2-(1'-cyclohexenyl)- and 2-nitro-2-(1'cycloheptenyl)-p-chlorophenyl-ethylene. Bul Ac Pol chim 6 no.5:313-318 '58. (EEAI 9:7)

1. Department of Organic Technology, Warsaw Technical University. Pharmaceutical Institute, Warsaw. Communicated by T.Urbanski.
(Nitrocyclohexenylchlorophenylethylene)
(Nitrocycloheptenylchlorophenylethylene)
(Absorption spectra)

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H

POLAND/Chemical Technology. Chemical Products and Their Applications. Pesticides.

Abs Jour : Ref Zhur-Khimiya, No 6, 1959, 20704

Author : Eckstein, Z., Halweg, H., Krakowka, P., Urbanski, T.

Inst : AS Poland.

Title : The Fungistatic Activity of 3,4-Dichlorophenoxyacethydroxamic Acid on Pathogenic Fungi in Vitro.

Orig Pub : Bull. Acad. polon. sci. Ser. sci. chim. geol. et geogr., 1958, 6, No 4, 235-238, XVIII

Abstract : Tests of the fungicidal activity of hydroxamic acids by the method of "cylinders" with Candida albicans 102, Cryptococcus neofor-

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POLAND/Chemical Technology. Chemical Products and Their Applications. Pesticides. H

Abs Jour : Ref Zhur-Khimiya, No 6, 1959, 20704

mans 38, Trichophyton gypseum 768, T. rubrum 3846, T. violaceum 3905, T. schoenleini III 1 F and Penicillium 45 showed that 2,4- and 2,5-dichlorphenoxy- and 2-methyl-4-chlorphenoxyacethydroxamic acids inhibit the growth of all tested species except C. albicans; alpha and beta-naphtoxyacethydroxamic acids suppresed the growth of fungi of the Trichophyton family; 5-nitro-2,4-dichlorphenoxyacethydroxamic acid is active against the latter three species; 2,4,6-trichlorphenoxyacethydroxamic acid is non-active. 3,4-dichlorphenoxyacethydroxamic acid (I) in concentrations of 0.005-0.25 ml/g suppresses the growth of

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POLAND/Chemical Technology. Chemical Products and Their Applications. Pesticides. H

Abs Jour : Ref Zhur-Khimiya, No 6, 1959, 20704

all the above-described fungi, as well as C. albicans K., C. tropicalis, C. krusei, Cryptococcus neoformans, Geotrichum I, G. malatensis 53, Histoplasma capsulatum. Sporotrichum shenskii, Nocardia asterides, N. madurae and Aspergillus fumigatus; in a concentration of 1 mg/ml, I suppresses the growth of the bacteria Mycobacterium 607, M. phlei, Bacillus Bodenheimer, B. circulaus, B. subtilis 219, B. subtilis 6633, B. subtilis 220, Escherichia coli, Serrata marscens 182, Staphylococcus aurens 209-P, S. oxford 11., Sarcina lutea. -- A. Grapov

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000411930007-6

EGKSTEIN,Z; SACHA,A.; SOBOTKS,W.; Urbanski,T.

On preparation and properties of 1-cyclooctenylnitromethane. Bul Ac Pol chim 6 no.10:621-624 '58. (KRAI 9:6)

1. Institute of Organic Synthesis, Polish Academy of Sciences. Institute of Pharmacy, Warsaw. Presented by T.Urbanski.

(Nitromethane) (Cyclooctene)
(Cyclooctanone) (Olefins)

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