NENCHUK, M.F.

Ancient carbonate rocks of the Rakhov Massif. Geol. zhur. 25 no.3:106-110 '65. (MIRA 18:11)

1. L'vovskiy gosudarstvennyy universitet im. I. Franko.

NENDTVICH, Andor

Let us make a spatial rearrangement for the most beautiful historical monuments of Pecsi. Pecsi musz szeml 8 no.4: 15-18 O-D'63.

S/169/61/000/011/013/065
D228/D304

AUTHORS: Kuznetsov, V.P., Kuz'mina, N.V., Nenelina, V.S.
Nersesov, I.L., Sultanova, Z.Z., and Kharin, D.A.

TITLE: Seismicity of the eastern part of the southern spurs of the Central Caucasus Range and some methodical questions of the study of seismicity of separate areas

PERIODICAL: Referativnyy zhurnal, Geofizika, no. 11, 1961, 18, abstract 11A162 (Izv. AN AzerbSSR, Ser. geol.-geogr. n., no. 5, 1960, 21 - 33)

TEXT: Determination of the degree of seismic activity on the southern spurs of the Central Caucasus Range was continued from the expeditional data of 1953 (for the first part see RZhGeofiz., no. 10, 1960, 11944) with a description of the strongest earthquakes: The Aksu-Kyurdamir earthquake of October 8, 1953, and the Avakhil earthquake of October 4, 1953 (the strongest ones); and the Caspian region earthquakes of August 8, September 14 and 19, and October 13. Epicentral zones - situated in a comparatively narrow strip along the Central Caucasus Range's southern slopes which follows the main
Card 1/3

structural directions - were considered. With the exception of some deviations, the seismically-active sections correspond to the transitional belt from the depressions to the mountain regions, i.e. the zone of contemporary contrasting movements. In the vicinity of Kutkashen a group of epicenters in a small area is situated transversely to the strike of the structures. Within the seismically-active belt the areas of epicenter concentration are separated by sections of complete quiescence. When comparing the expeditional data of 1953 and 1951 - 1952 with those of the network of permanent stations for the period from 1913, it is established that a certain redistribution of seismic activity has taken place, although the locations of strong earthquakes coincide with areas which are distinguished by their activity according to the observations of seasonal expeditions. The expeditional investigations enable observational data to be processed more accurately and a better basis to be constructed for the relations of seismic and tectonic phenomena. The complexity of the geologic structure of the study area hampered the obtaining of the coordinates of earthquake foci with the required precision. The use of different methods permitted determination of the epicenter positions with an accuracy of up to ± 5 km, and also

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Seismicity of the eastern part ...

the propagational velcoties of seismic waves and their ratios. The ratio of the velocities for different foci varied from west to east from 1.8 (the Vartashen district) to 2.2 (the Avakhil district) evidently because of the presence of a thick series of sedimentary rocks in the eastern areas. The low value of the fictitious velocity, which varies from 4.1 (Astrakhanovka) to 6.1 km/sec. (Durukhsha) is a consequence of the low value of the velocity ratio. [Abstractor's note: Complete translation].

Card 3/3

HARSING, Laszlo; KOVER, Gyorgy; DUBECZ, Erzsebet; NENES, Gyorgy

Effect of hypothermia on the inulin space. Kiserletes orvostud. 13 no.4:381-384 Ag '61.

1. Budapesti Orvostudomanyi Egyetem Elettani Intezete.

(BODY TEMPERATURE) (INULIN metab)

68-58-3-11/22

AUTHORS: Nenich, V.N., Drinfel'd, P.I., Tselykovskaya, N.K. and Pristavko, F.I.

TITLE: Effluents from the Indene-Coumarone Resin Plant and Possibilities of Their Purification (Stochnyye vody tsekha Inden-Kumaronovykh smol i vozmozhnosti ikh obezvrezhivaniya)

PERIODICAL: Koks i Khimiya, 1958, Nr 3, pp 40 - 44 (USSR).

ABSTRACT: Biological treatment of coke oven effluents deteriorated when the effluent from the Indene-Coumarone Resin Plant was added . Methods of pre-treatment of this effluent were investigated. It was found that the best results are obtained when the neutralised effluent is passed through a vacuo-filter in order to separate aluminium hydroxide (derived from aluminium chloride, the catalyst used for polymerisation), then into a settling tank for the separation of benzole. After the separation of benzole, the effluent is passed into the biological treatment tank. At present, an installation based on the above scheme (Fig.5) is being designed. There are 4 tables and 5 figures.

ASSOCIATION: Kadiyevskiy koksokhimicheskiy zavod (Kadiyevka Coke Oven Works)

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SOV/68-58-10-13/25

AUTHORS: Yastrzhem'skaya, O.V., Andreyeva, V.S., Nenich, V.N., Royter, M.K., Drinfel'd, P.Ye., and Bilym, L.M.

TITLE: From Experience of Putting the Indene-coumarone Resin Plant on the Kadiyevka Coking Works into Operation (Opyt puska i raboty tsekha inden-kumaronovykh smol na Kadiyevskom koksokhimicheskom zavode)

PERIODICAL: Koks i Khimiya, 1958, Nr 10, pp 40 - 44 (USSR)

ABSTRACT: The plant was put into operation in 1955. The scheme of the operation of the plant as designed is shown in Figure 1 and changes introduced are shown in Figures 2 and 3. Aluminium chloride is used as a catalyst in a proportion of 0.35% of the raw material. The polymerisation process begins at 20 - 30 °C and is finished at 110 °C. The main difficulties were encountered in the distillation plant due to the incorrect design of the evaporators and due to an excessive corrosion of the condenser. All resin pipe-lines were found to be too long and complicated. Cooling drums for resin were insufficient. The initial losses of hydrocarbons amounted to 18-20% and were reduced (by unspecified methods) to

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SOV/68-58-10-13/25

From Experience of Putting the Indene-coumarone Resin Plant on the Kadiyevka Coking Works into Operation

6-8%. Softening temperature of the resin produced 100 - 110 °C. It is pointed out that in order to decrease corrosion, an enamelled distillation apparatus and a reactor for the preparation of aluminium chloride complex should be introduced. There are 3 figures.

ASSOCIATIONS: UKhIN and Kadiyevskiy koksokhimicheskiy zavod (Kadiyevka Coking Works)

Card 2/2

NENICH, V.N.; DRINFEL'D, P.I.; TSELYKOVSKAYA, N.K.; DAKHNENKO, N.Ya.

Dephenolization of waste waters from recovery plants by the "microbe method." Koks i khim. no.1:38-41 '60. (MIRA 13:6)

1. Kadiyevskiy koksokhimicheskiy zavod.
(Kadiyevka--Sewage disposal)
(Phenols)

FD-1702

USSR/Geophysics - Earthquake focus

NENILINA, V. S.

Card 1/1 : Pub. 45-2/12

Author : Savarenskiy, Ye. F., and Nenilina, V. S.

Title : Taking account of geological inhomogeneities in the determination of the position of source (focus) of earthquakes

Periodical : Izv. AN SSSR, Ser. geofiz., 17-30, Jan-Feb 1955

Abstract : The authors discuss methods for determining the position of earthquake foci under the condition of establishing the velocities within plane-parallel horizontal layers of the terrestrial crust. They propose a method for taking into account the geological inhomogeneities. This method is applied to certain observations carried out in 1951 by stations of the regional type of the Aralo-Caspian expedition, Academy of Sciences USSR. Four references (e.g. S. I. Golenetskiy and A. A. Treskov, "Method of isochrones," Trudy Geofiz. in-ta AN SSSR, No 21 (148), 1953; "Method of hypocenters," ibid., No 14 (141), 1952).

Institution : Geophysics Institute, Academy of Sciences USSR

Submitted : January 25, 1954

BAGDASAROVA, A.M.; ISLAMOV, K.Sh.; KORIDALIN, Ye.A.; KUZNETSOV, V.P.; KUZ'MINA, N.V.; NENILINA, V.S.; NERSESOV, I.L.; SULTANOVA, Z.Z.; KHARIN, D.A.

Seismicity of the eastern part of the southern spurs of the Greater Caucasus and some problems of methodology in studying the seismicity of individual regions. Report No.1. Izv.AN Azerb.SSR. Ser.geol.-geog.nauk no.6:121-131 '59. (MIRA 15:4)
(Caucasus--Seismology)

BAGDASAROVA, A.M.; ISLAMOV, K.Sh.; KORIDALIN, Ye.A.; KUZNETSOV, V.P.; KUZ'MINA, N.V.; NENILINA, V.S.; NERSESOV, I.L.; SULTANOVA, Z.Z.; KHARIN, D.A.

Seismicity of the eastern part of the southern spurs of the Greater Caucasus and some problems of methodology in studying the seismicity of individual regions. Report No.3. Izv.AN Azert.SSR. Ser.geol.-geog.nauk i nefti. no.4:13-24 '61. (MIRA 15:1)
(Caucasus--Seismology)

CIBIRAS, P., kand. med. nauk; DAKTARAVICIENE, E., kand. med. nauk; JARZEMSKAS, J., kand. med. nauk [deceased]; JOCEVICIENE, A., kand. med.nauk; KRIKSTOPAITIS, M., kand. med. nauk; NENISKIS,J., kand. med. nauk; STEPONAITIENE, L., kand. med. nauk; SURKUS, J., kand. med. nauk; SIIMANAS, S., kand. biolog. nauk; CEPULIS, St., prof.; KUPCINSKAS, J., prof.; LASAS, Vl., prof.; SIDERAVICIUS,Br., prof.; KANOPKA, E.,dots.; KVIKLYS, V.,dots.; LABANAUSKAS, K., dots.; POLUKORDAS, H., dots.; BABUBLYS, P., doktor; CAPKEVICIUS,V., doktor; MAKARIUNAS, P., doktor; PAKONAITIS, P., doktor; STUOKA.R., doktor; SURGAILIS, H., doktor; PAULIUKONIENE, J., red.; ANAITIS,J., tekhn. red.

[Health and diseases] Antrasis pataisytas leidimas. Vilnius, Valstybine politines ir mokslines literaturos leidykla, 1961. 356 p.
(MIRA 15:3)

(HYGIENE) (PATHOLOGY)

SIMKUNAS, V.; MASLAUSKIENE, M.; ZAIKAUSKAS, M., red.; ZILINSKIENE,V., red.; KVIKLYS, V., kand. med. nauk, red.; MOTIEJUNAS, L., kand. med. nauk, red.; NEMISKIS, J., kand. med. nauk, red.; STECOVSKIS, A.L., tekhn. red.

[Lithuanian medical bibliography] Lietuviskoji medicinine bibliografija. Vilnius, Valstybine moksline medicinos biblioteka. Vol.2. 1958-1960. 543 p. (MIRA 17:3)

1. Predsedatel' Soveta Gosudarstvennoy nauchno-meditsinskoy biblioteki Ministerstva zdravookhraneniya Litovskoy SSR (for Zaikauskas). 2. Direktor Gosudarstvennoy nauchno-meditsinskoy biblioteki Ministerstva zdravookhraneniya Litovskoy SSR (for Zilinskiene).

NENISKIS, J.

Apropos of lowering the number of abortions performed outside of hospitals. Sveik. apsaug. 9 no.1:35-38 Ja'64.

1. LTSR Sveikatos apsaugos ministerijos vyr. akuseris-ginekologas.

*

IONITA, Gheorghe, ing. (Bucuresti); NENITA, Eugen, ing. (Bucuresti)

Automation of hydroelectric power stations in Rumania. Energetica Rum 10 no.7:302-310 Jl '62.

1. Proiectant sef la Institutul de studii si proiectari energetice (for Ionita). 2. Proiectant la Institutul de studii si proiectari energetice (for Nenita).

1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

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Derivatives of 2-methyl-5-hydroxyindole. Costin D. Nenitzescu. *Bull. soc. chim. Romania* 11, 37-43(1929); cf. *C. A.* 23, 2716.—Condensations with quinone (I) have been reported previously (*Ber.* 21, 3005; *J. prakt. Chem.* 45, 78; *C. A.* 22, 1148). I (44 g.) and 52 g. of Me(H$_2$N)C:CHCO$_2$Et (II) in 120 cc. Me$_2$CO after heating 1 hr. yield 23 g. of 2-methyl-3-carbethoxy-5-hydroxyindole (III), m. 265°. With 2 mols. of II in C$_6$H$_6$ a compd., C$_{22}$H$_{24}$O$_6$N$_2$, m. 173°, results. III with NaOH yields 2-methyl-5-hydroxyindole, m. 133°, and with Me$_2$SO$_4$ 2-methyl-3-carbethoxy-5-methoxyindole (IV), m. 161°, which with NaOH yields the acid, m. 208°, which on melting gives 2-methyl-5-methoxyindole (V), m. 89-90°. Condensation as above with Me(NHPh)C:CHCO$_2$Et gives 1-phenyl-2-methyl-3-carbethoxy-5-hydroxyindole, m. 205-6° (alc.) and with glycine-acetoacetic ester (*Ber.* 34, 437), Et [2-methyl-3-carbethoxy-5-hydroxyindyl-1] acetate, m. 148°. II (1.6 g.), 1 cc. HC(OEt)$_3$ and 3 cc. of Ac$_2$O yield on heating tri[2-methyl-5-methoxyindyl]methane, m. 227°. V (2 g.) in 20 cc. HCO$_2$H satd. with HCl yields [2-methyl-5-methoxyindyl][2-methyl-5-methoxyindolenyl]methene·HCl, m. 230° (decompn.). V. F. Harrington

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1ST AND 2ND ORDERS

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3RD AND 4TH ORDERS

Some reactions of magnesylpyrroles and indoles. Costin D. Nenitzescu. *Bul. soc. chim. Romania* 11, 130-4(1930).—The magnesyl radical of the pyrrole derivs. is shown to be attached to C rather than to N since the products give a violet color with Michler's ketone (cf. Gilman and Heck, *C. A.* 23, 1000). The magnesylindole derivs. give no color and are thus *N*-magnesyl derivs. EtMgBr and 3,5-dimethylpyrrole in Et_2O gave a soln. of magnesylpyrrole to which *N*-formylpiperidine was slowly added. The resulting soln. was treated with concd. NH_4Cl soln., washed with H_2O and evapd., yielding *bis[3,5-dimethylpyrrole-2]-methylpiperidine*, m. 133° (from MeOH), which, when warmed with concd. HCl, gave *bis[3,5-dimethylpyrrole-2] methene*, m. 117° (from alc.), whose *HCl salt* m. 227° (darkens 210°). T. H. Rider

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CA

A modification of the Gattermann method for the preparation of phenol- and pyrrole-aldehydes. Costin D. Nenitzescu and Dumitru Isacescu. *Bul. soc. chim. Romania* 11, 125-9(1930).—Formamide (I) and $POCl_3$ (II) are used instead of HCN and HCl as a source of HClC:NH. Resorcinol (III) and I in anhyd. Et_2O were treated with II, yielding a cryst. *compd.* which was heated with water, yielding *III-aldehyde*, m. 134-6° (from H_2O). In the same manner were prepd.: *3,5-dimethylformylpyrrole*, m. 90° (from H_2O); *2,5-dimethyl-3-carbethoxy-4-methenylaniline-pyrrole.HCl*, m. 221° (from H_2O), which with NH_4OH gave *3,5-dimethyl-3-carbethoxy-4-formylpyrrole*, m. 151°; and *3,5-dimethyl-2-carbethoxy-4-formylpyrrole*, which when warmed with H_2O gave the *aldehyde*, m. 145°.

T. H. Rider

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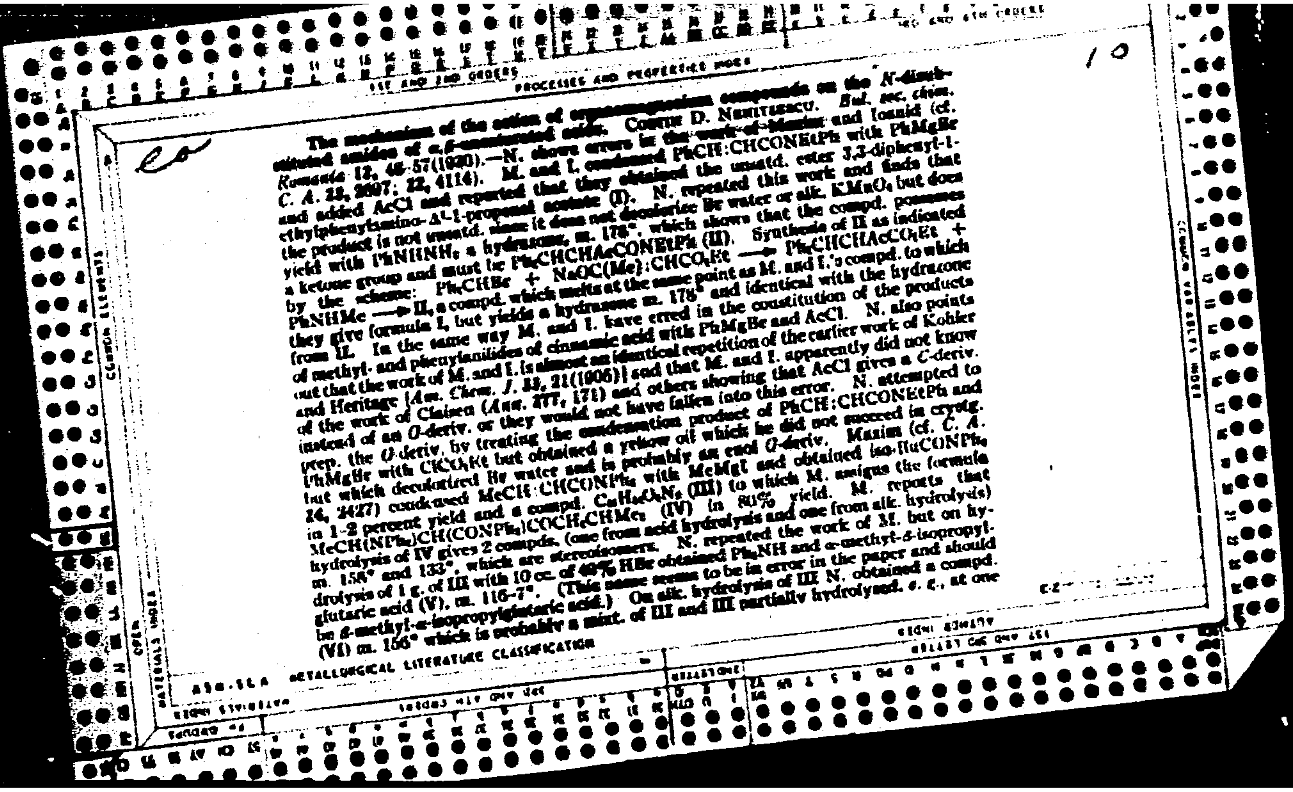

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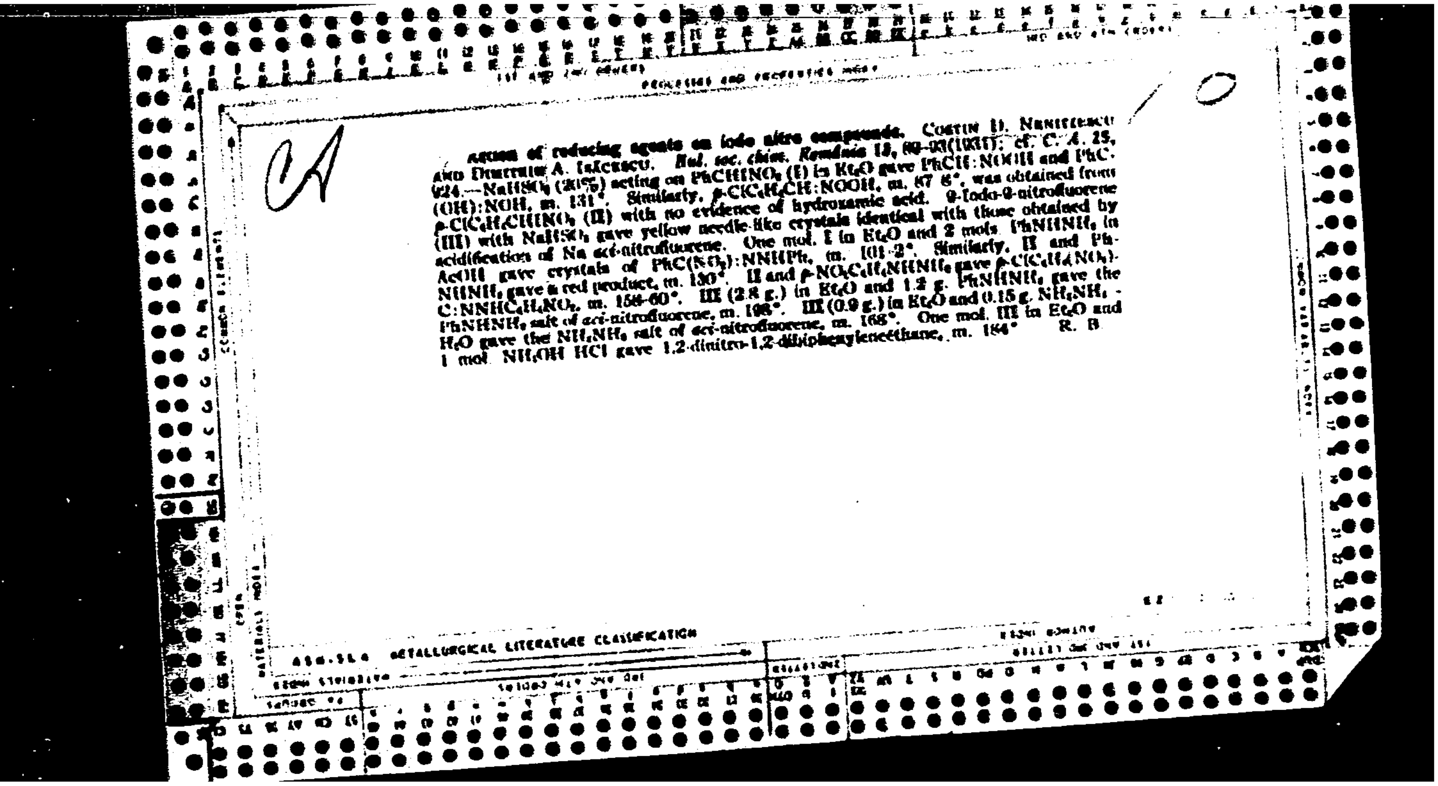
ea

The mechanism of the action of organomagnesium compounds on the N-disubstituted amides of α,β-unsaturated acids. Costin D. Nenitzescu. *Bul. soc. chim. Romania* 12, 48-57(1930).—N. shows errors in the work of Maxim and Ioanid (cf. *C. A.* 23, 2007; 22, 4114). M. and I. condensed PhCH:CHCONEtPh with PhMgBr and added AcCl and reported that they obtained the unsatd. ester 3,3-diphenyl-1-ethylphenylamino-$\Delta^{1,1}$-propenol acetate (I). N. repeated this work and finds that the product is not unsatd. since it does not decolorize Br water or alk. $KMnO_4$ but does yield with $PhNHNH_2$ a hydrazone, m. 178°, which shows that the compd. possesses a ketone group and must be $Ph_2CHCHAcCONEtPh$ (II). Synthesis of II as indicated by the scheme: $Ph_2CHBr + NaOC(Me):CHCO_2Et \longrightarrow Ph_2CHCHAcCO_2Et + PhNHMe \longrightarrow$ II, a compd. which melts at the same point as M. and I.'s compd. to which they give formula I, but yields a hydrazone m. 178° and identical with the hydrazone from II. In the same way M. and I. have erred in the constitution of the products of methyl- and phenylanilides of cinnamic acid with PhMgBr and AcCl. N. also points out that the work of M. and I. is almost an identical repetition of the earlier work of Kohler and Heritage [*Am. Chem. J.* 33, 21(1906)] and that M. and I. apparently did not know of the work of Claisen (*Ann.* 277, 171) and others showing that AcCl gives a *C*-deriv. instead of an *O*-deriv. or they would not have fallen into this error. N. attempted to prep. the *O*-deriv. by treating the condensation product of PhCH:CHCONEtPh and PhMgBr with $ClCO_2Et$ but obtained a yellow oil which he did not succeed in crystg. but which decolorized Br water and is probably an enol *O*-deriv. Maxim (cf. *C. A.* 24, 2427) condensed $MeCH:CHCONPh_2$ with MeMgI and obtained iso-$BuCONPh_2$ in 1-2 percent yield and a compd. C[illegible]H[illegible]O_2N_2 (III) to which M. assigns the formula $MeCH(NPh_2)CH(CONPh_2)COCH_2CHMe_2$ (IV) in 80% yield. M. reports that hydrolysis of IV gives 2 compds. (one from acid hydrolysis and one from alk. hydrolysis) m. 158° and 133°, which are stereoisomers. N. repeated the work of M. but on hydrolysis of 1 g. of III with 10 cc. of 48% HBr obtained Ph_2NH and α-methyl-δ-isopropylglutaric acid (V), m. 116-7°. (This name seems to be in error in the paper and should be β-methyl-α-isopropylglutaric acid.) On alk. hydrolysis of III N. obtained a compd. (VI) m. 166° which is probably a mixt. of III and III partially hydrolyzed, *e. g.*, at one of the CO_2H groups. Treatment of VI with 15 cc. 40% HBr yielded V. N. attributes the errors of M. to incomplete hydrolysis of III. N. explains the formation of III by the following scheme and shows its constitution to be represented by VII rather than IV. $MeCH:CHCONPh_2 + MeMgI \longrightarrow Me_2CHCH:C(OMgI)NPh_2$ (VIII) + $MeCH:CHCONPh_2 \longrightarrow Me_2CHC(CHMeCH_2CONPh_2):C(OMgI)NPh_2 + H_2O \longrightarrow Me_2CHCH(CHMeCH_2CONPh_2)CONPh_2$ (VII) + MgIOH. VIII possesses enolic properties and adds on to a 2nd mol. of $MeCH:CHCONPh_2$. The mechanism of addn. of organomagnesium compds. to *N*-disubstituted amides of both cinnamic and crotonic acids is at the ends of the conjugated system C:C.C:O and in accord with the work of Kohler and Heritage.

Owen K. Sheppard



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Action of reducing agents on iodo nitro compounds. Costin D. Nenitzescu and Dumitru A. Isacescu. *Bul. soc. chim. Romania* 13, 89-93(1931); cf. *C. A.* 25, 924.—NaHSO$_3$ (20%) acting on PhCHINO$_2$ (I) in Et$_2$O gave PhCH:NOOH and PhC(OH):NOH, m. 131°. Similarly, *p*-ClC$_6$H$_4$CH:NOOH, m. 87-8°, was obtained from *p*-ClC$_6$H$_4$CHINO$_2$ (II) with no evidence of hydroxamic acid. 9-Iodo-9-nitrofluorene (III) with NaHSO$_3$ gave yellow needle-like crystals identical with those obtained by acidification of Na *aci*-nitrofluorene. One mol. I in Et$_2$O and 2 mols. PhNHNH$_2$ in AcOH gave crystals of PhC(NO$_2$):NNHPh, m. 101-2°. Similarly, II and PhNHNH$_2$ gave a red product, m. 130°. II and *p*-NO$_2$C$_6$H$_4$NHNH$_2$ gave *p*-ClC$_6$H$_4$(NO$_2$)C:NNHC$_6$H$_4$NO$_2$, m. 158-60°. III (2.8 g.) in Et$_2$O and 1.2 g. PhNHNH$_2$ gave the PhNHNH$_2$ salt of *aci*-nitrofluorene, m. 198°. III (0.9 g.) in Et$_2$O and 0.15 g. NH$_2$NH$_2$.H$_2$O gave the NH$_2$NH$_2$ salt of *aci*-nitrofluorene, m. 168°. One mol. III in Et$_2$O and 1 mol. NH$_2$OH.HCl gave 1,2-dinitro-1,2-diphenyleneethane, m. 184°. R. B.

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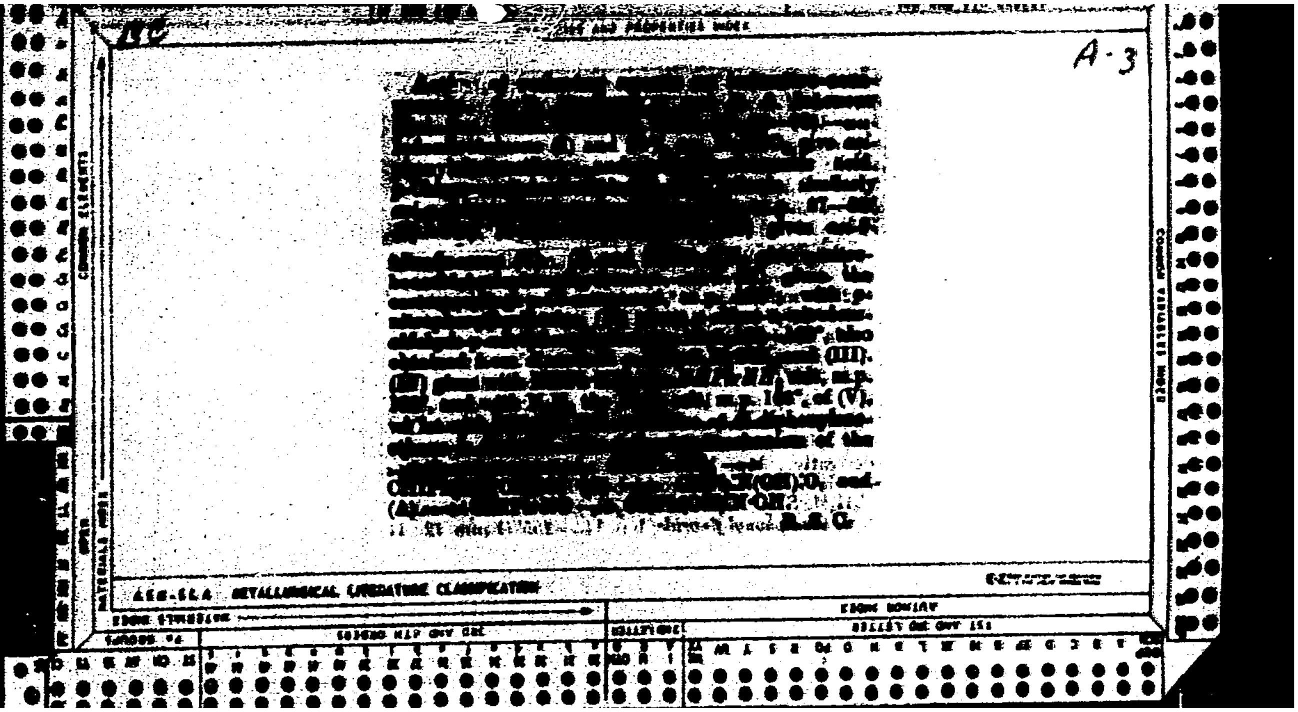

A-3

[illegible] (III). [illegible] m.p. [illegible] m.p. 1[illegible]° of (V). [illegible] of the [illegible] (OH)$_2$O, and (Al[illegible] OH. [illegible] R. G.

METALLURGICAL LITERATURE CLASSIFICATION

A-3

aci-Nitro-compounds. IV. Mechanism of conversion of nitro-derivatives into hydroxamic acids. C. D. [illegible] and D. A. Isacescu (Bul. Soc. chim. [illegible], 1932, 14, [illegible]—68).—Interaction of [illegible] (I) or of its Na salt with HCl in dry Et_2O gives benzhydroxamic chloride, with intermediate formation of a blue coloration. The transformation is explained as follows: $CHPh:NO\cdot OH + HCl \longrightarrow [Cl\cdot CHPh\cdot NH(:O)\cdot OH] \xrightarrow{-H_2O} Cl\cdot CHPh\cdot NO$ (blue) $\longrightarrow Cl\cdot CPh:N\cdot OH$. With acid chlorides (AcCl, BzCl, $POCl_3$) (I) gives the same product, but its Na salt gives the acyl derivative of the hydroxamic acid, *e.g.*, $CPh(OH):N\cdot OAc$. The Na salt of 9-acinitrofluorene (II), however, gives with AcCl in Et_2O at 0° a stable, yellow *Ac* derivative, $C_{12}H_8:CNO\cdot OAc$, m.p. 99—100°, and similarly, a *Bz* derivative, m.p. 134—135°; the former is also obtained from (II) and keten at about −70°. With (I) keten gives an unstable *Ac* derivative, m.p. 60° (diffuse), decomposed by warm H_2O to acetylbenzhydroxamic acid. The *Me* ether of (II), $C_{12}H_8:CNO\cdot OMe$, m.p. 86° (decomp. into CH_2O and fluorenoneoxime), is prepared from its K salt and Me_2SO_4. H. A. P.

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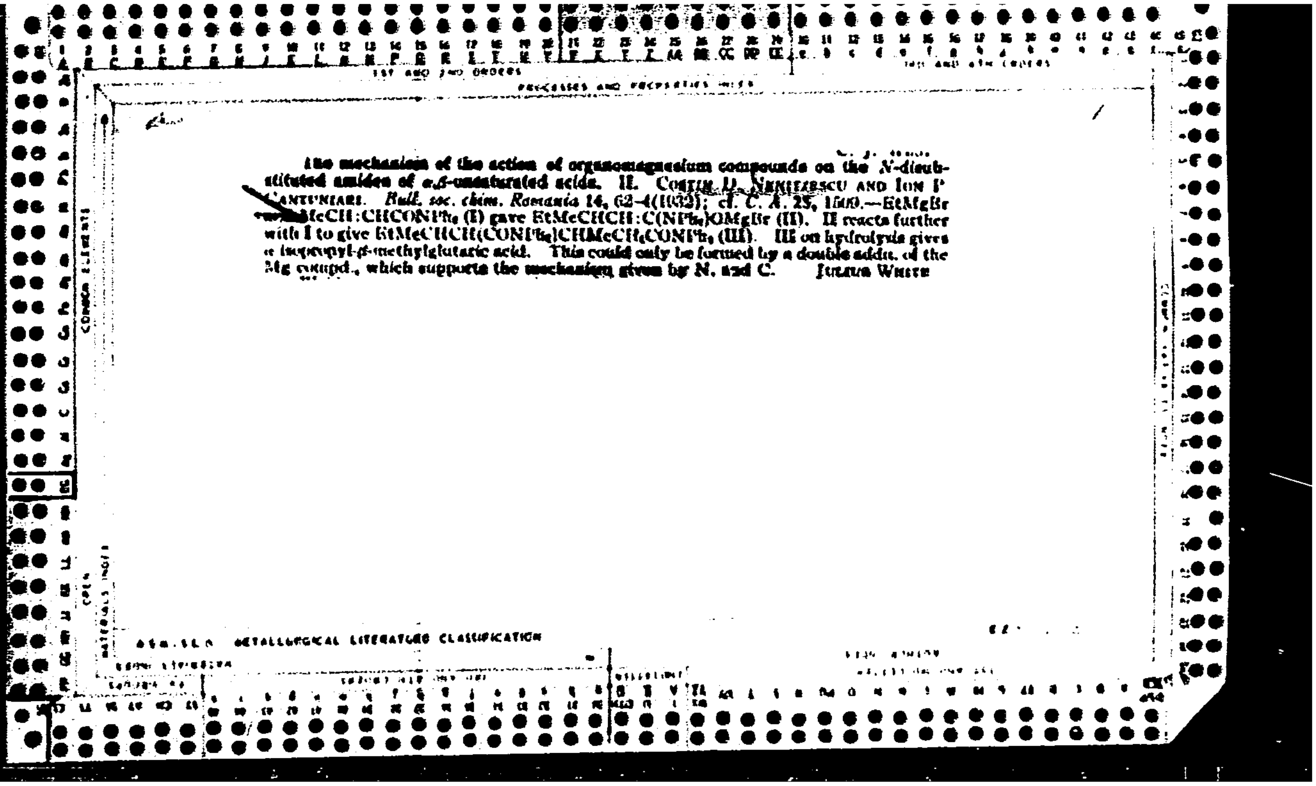
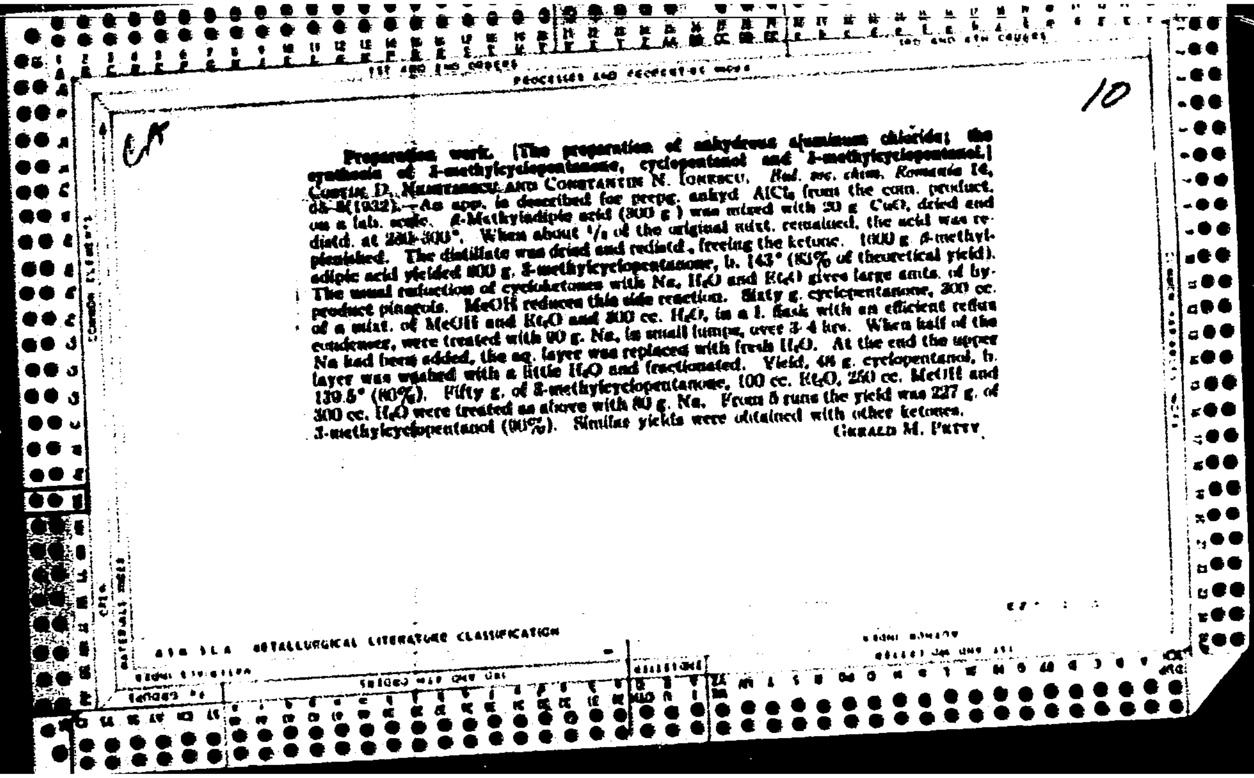


The mechanism of the action of organomagnesium compounds on the *N*-disubstituted amides of α,β-unsaturated acids. II. Costin D. Nenitzescu and Ion P. Cantuniari. *Bull. soc. chim. Romania* 14, 62-4(1932); cf. *C. A.* 25, 1500.—EtMgBr with MeCH:CHCONPh$_2$ (I) gave EtMeCHCH:C(NPh$_2$)OMgBr (II). II reacts further with I to give EtMeCHCH(CONPh$_2$)CHMeCH$_2$CONPh$_2$ (III). III on hydrolysis gives α-isopropyl-β-methylglutaric acid. This could only be formed by a double addn. of the Mg compd., which supports the mechanism given by N. and C. Julian White



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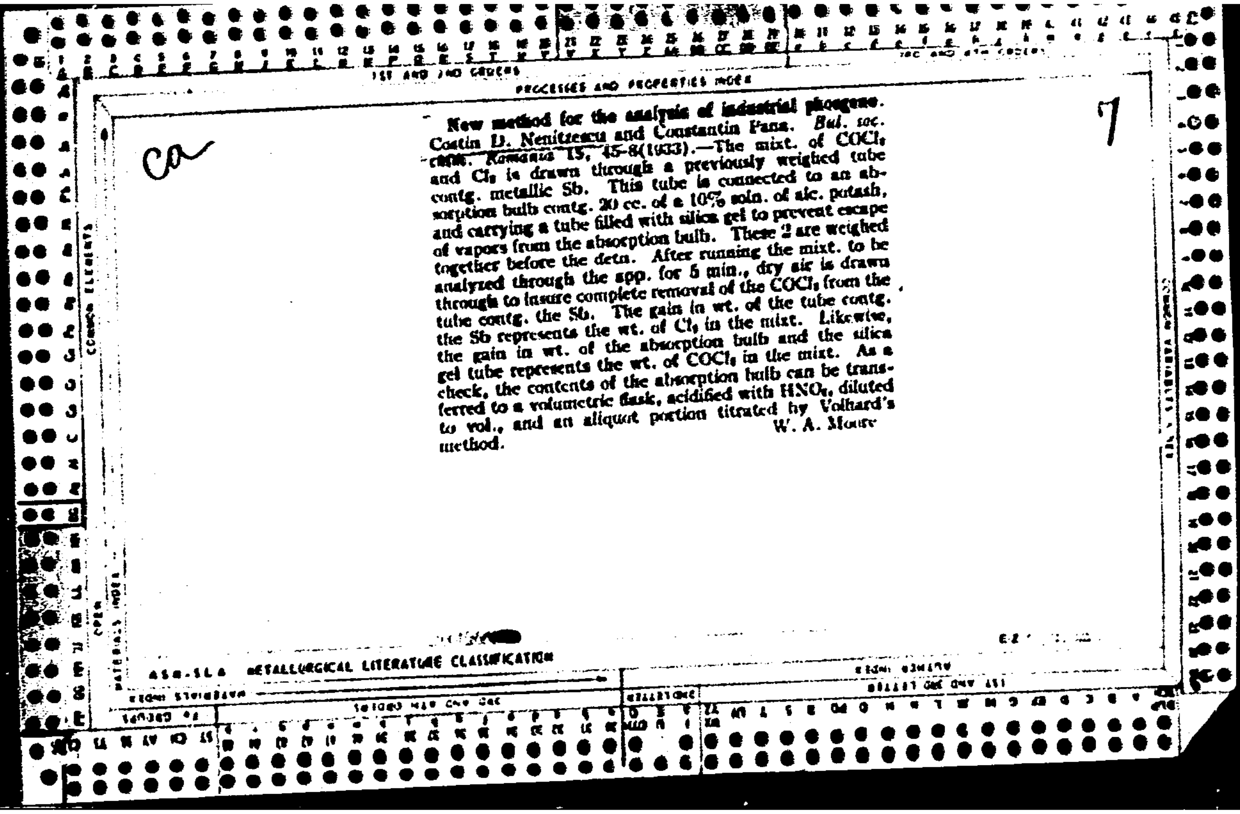
Preparation work. [The preparation of anhydrous aluminum chloride; the synthesis of 3-methylcyclopentanone, cyclopentanol and 3-methylcyclopentanol.] Costin D. Nenitzescu and Constantin N. Ionescu. *Bul. soc. chim. Romania* 14, 53-8(1932).—An app. is described for prepg. anhyd. $AlCl_3$ from the com. product, on a lab. scale. β-Methyladipic acid (800 g.) was mixed with 20 g. CuO, dried and distd. at 280-300°. When about 1/6 of the original mixt. remained, the acid was replenished. The distillate was dried and redistd., freeing the ketone. 1000 g. β-methyladipic acid yielded 400 g. 3-methylcyclopentanone, b. 143° (81% of theoretical yield). The usual reduction of cycloketones with Na, H_2O and Et_2O gives large amts. of by-product pinacols. MeOH reduces this side reaction. Sixty g. cyclopentanone, 300 cc. of a mixt. of MeOH and Et_2O and 300 cc. H_2O, in a 1. flask with an efficient reflux condenser, were treated with 90 g. Na, in small lumps, over 3-4 hrs. When half of the Na had been added, the aq. layer was replaced with fresh H_2O. At the end the upper layer was washed with a little H_2O and fractionated. Yield, 48 g. cyclopentanol, b. 139.5° (80%). Fifty g. of 3-methylcyclopentanone, 100 cc. Et_2O, 250 cc. MeOH and 300 cc. H_2O were treated as above with 80 g. Na. From 5 runs the yield was 227 g. of 3-methylcyclopentanol (90%). Similar yields were obtained with other ketones.

Gerald M. Petty



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New method for the analysis of industrial phosgene. Costin D. Nenitzescu and Constantin Pana. *Bul. soc. chim. Romania* 15, 45-8(1933).—The mixt. of $COCl_2$ and Cl_2 is drawn through a previously weighed tube contg. metallic Sb. This tube is connected to an absorption bulb contg. 20 cc. of a 10% soln. of alc. potash, and carrying a tube filled with silica gel to prevent escape of vapors from the absorption bulb. These 2 are weighed together before the detn. After running the mixt. to be analyzed through the app. for 5 min., dry air is drawn through to insure complete removal of the $COCl_2$ from the tube contg. the Sb. The gain in wt. of the tube contg. the Sb represents the wt. of Cl_2 in the mixt. Likewise, the gain in wt. of the absorption bulb and the silica gel tube represents the wt. of $COCl_2$ in the mixt. As a check, the contents of the absorption bulb can be transferred to a volumetric flask, acidified with HNO_3, diluted to vol., and an aliquot portion titrated by Volhard's method. W. A. Moore

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

PROCESSES AND PROPERTIES INDEX

BC

a-3

Cannizzaro reaction. C. D. Nenitzescu and I. Gavăt (Bul. Soc. Chim. România, 1934, 16, [A], 45—46).—The velocity of the Cannizzaro reaction with PhCHO in 80% MeOH at 30° is greatly increased by increase in the concn. of KOH from 0·5N to 4·0N and slightly (catalytically) by Cu at 50°. $NMe_3Et\cdot OH$ affects the reaction, but Na_2CO_3 and Na_2HPO_4 do not. Equimol. mixtures of PhCHO with CH_2O or $OMe\cdot C_6H_4\cdot CHO$ give both pairs of acids and alcohols. A large excess of CH_2O gives almost exclusively HCO_2H and the aromatic alcohol ($CH_2Ph\cdot OH$, $OMe\cdot C_6H_4\cdot CH_2\cdot OH$, or m-$NO_2\cdot C_6H_4\cdot CH_2\cdot OH$). R. S. C.

METALLURGICAL LITERATURE CLASSIFICATION

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

13

New methods for the manufacture of yperite. Costin D. Nenitzescu and Nicolae Scarlatescu. *Antigaz* (Bucharest) 9, No. 9/10, 12-21; No. 11/12, 3-11(1935); *Chem. Zentr.* 1936, I, 4240.—The oxol method as well as the process of Guthrie-Niemann is discussed and their disadvantages are explained. In the present investigation the *thiodiglycol* was obtained by condensation of ethylene oxide with H_2S without the addn. of a catalyst. However, slight amts. of thiodiglycol had to be added which appeared to function as a solvent for both gases. An appropriate app. for carrying out the reaction is described. The reaction is exothermic and the temp. should be maintained at 40-60° by cooling, since below 40° the yield is reduced and above 60° the product is brown and has an objectionable odor suggesting decompn. and side reactions. The max. yield obtained was 90.35%. The yperite prepd. by the action of concd. HCl on the thiodiglycol so obtained is only faintly yellow; the raw product has only a faint odor and solidifies at 12°. Raw materials required by this new method are petroleum fractions (for the prepn. of the C_2H_4), lime, Fe sulfide, S and HCl. If the C_2H_4 is prepd. from alc., the latter is required as a raw material, as is Cl if the C_2H_4O is prepd. from $Cl(CH_2)_2OH$. This new process has the advantages of yielding a purer product which keeps better than that obtained by the oxol method, requiring simpler app. than the latter method and being less dangerous. The main step in the process, the prepn. of the dithioglycol, can be carried out as a continuous operation.

W. A. Moore

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

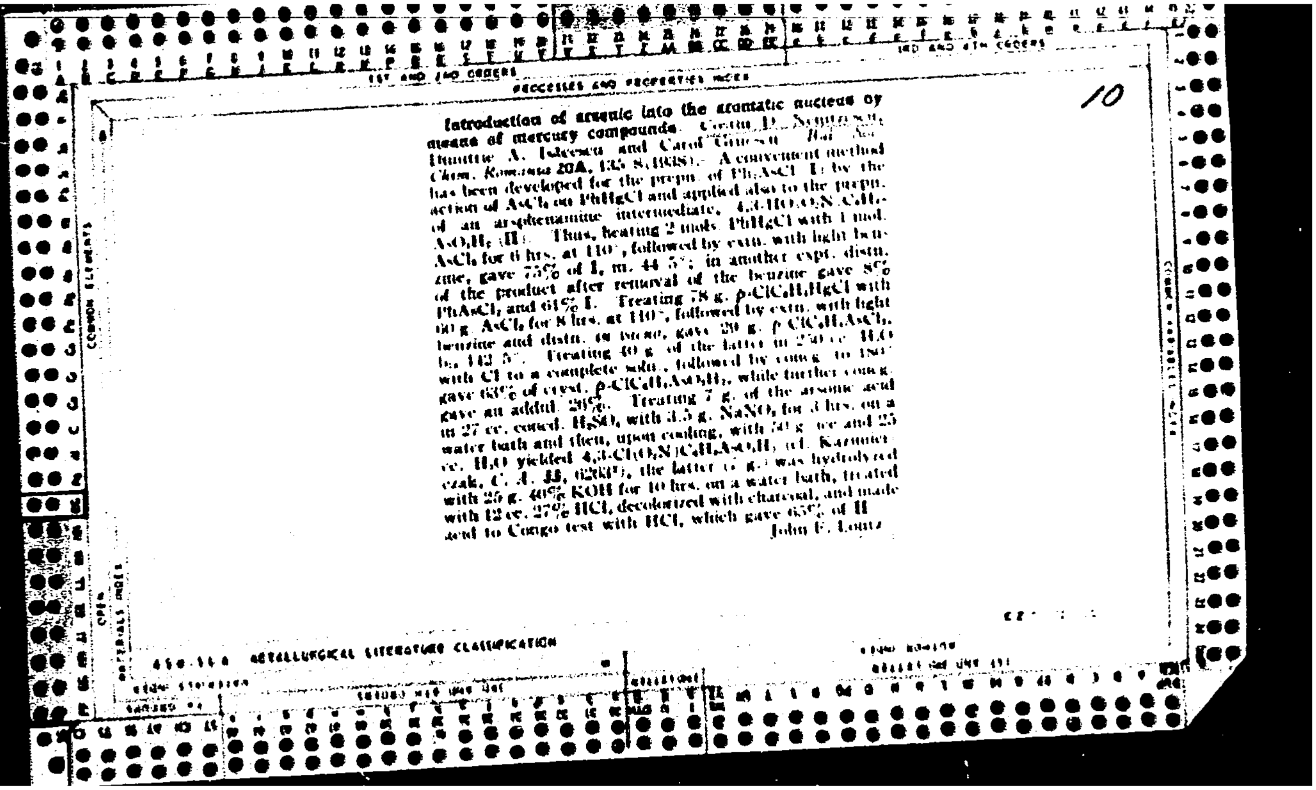
3RD AND 4TH ORDERS

10

Mercuration of benzene and chlorobenzene. Costin D. Nenitzescu, Dumitrie A. Isăcescu and Carol Gruescu. *Bul. Soc. Chim. România* 20A, 127-34(1938).—The introduction of Hg as the acetate into the C_6H_6 nucleus is reviewed briefly along with consideration of Maynard's (C. A. 18, 2141) proposed equil. reaction, C_6H_6 + Hg(OAc)$_2$ ⇌ PhHgOAc (I) + AcOH, to which some exception is taken in view of the inability to duplicate the high yield through the use of EtOH for the removal of the AcOH component of the equil. and the formation of polymercurated products. The present study was accordingly directed toward ascertaining empirically the proportions of Hg(OAc)$_2$, C_6H_6 and AcOH which would give the highest yields of I. for this purpose the detn. of I in the presence of Hg(OAc)$_2$ was based upon the large difference in the solubilities of these 2 compds. in dry C_6H_6. The expts. were made with 10 g. (1/30 mol.) Hg(OAc)$_2$ using 1, 10, 20, 25, 35 and 45 mol. ratios of C_6H_6 and 5, 10, 15, 20, 30 and 40 mol. ratios of glacial AcOH, with 3, 6 and 9 hrs. of heating; the results are tabulated and indicated diagrammatically. It was observed that the larger the amt. of AcOH, the quicker the Hg(OAc)$_2$ goes into soln.; the smaller the amt. of C_6H_6 and the greater the amt. of AcOH, the greater is the amt. of polymercurated compds. The yield of I increases with excess C_6H_6 and with the AcOH up to a max. between 10-20 mols., beyond which it drops off; it also increases with time up to a max. and then declines. The optimum condition for the prepn. of I consists of warming 160 g. Hg(OAc)$_2$ with 137 g. C_6H_6 and 600 g. AcOH for 9 hrs. on the water bath, removing the excess AcOH and C_6H_6 under diminished pressure and extg. the reaction mixt. (166 g.) in a Soxhlet app., which gave a 155 g. (92%) yield of I; for conversion to PhHgCl, 100 g. of I was heated with 200 cc. 25% KCl with stirring for 5 hrs. on the water bath, yielding 98 g. of product, m. 240°. Applying this reaction to PhCl, a 50% yield of p-ClC_6H_4HgOAc (II) (cf. Hanke, C. A. 17, 2275), m. 194°, from MeOH, was obtained, and the product was identified by converting to p-ClC_6H_4Br with H_2O and Br. On account of the difficulty in wetting II with H_2O it was not possible to apply the above conversion to the chloride, however, by treating 220 g. II with 500 cc. of a mixt. of 2 parts AcOH and 1 part 6% KCl soln. a 190-g. yield of p-ClC_6H_4HgCl (cf. Nesmeyanov and Kocsh, C. A. 28, 6429), m. 240°, from Me_2CO, was obtained.

John F. Lowe

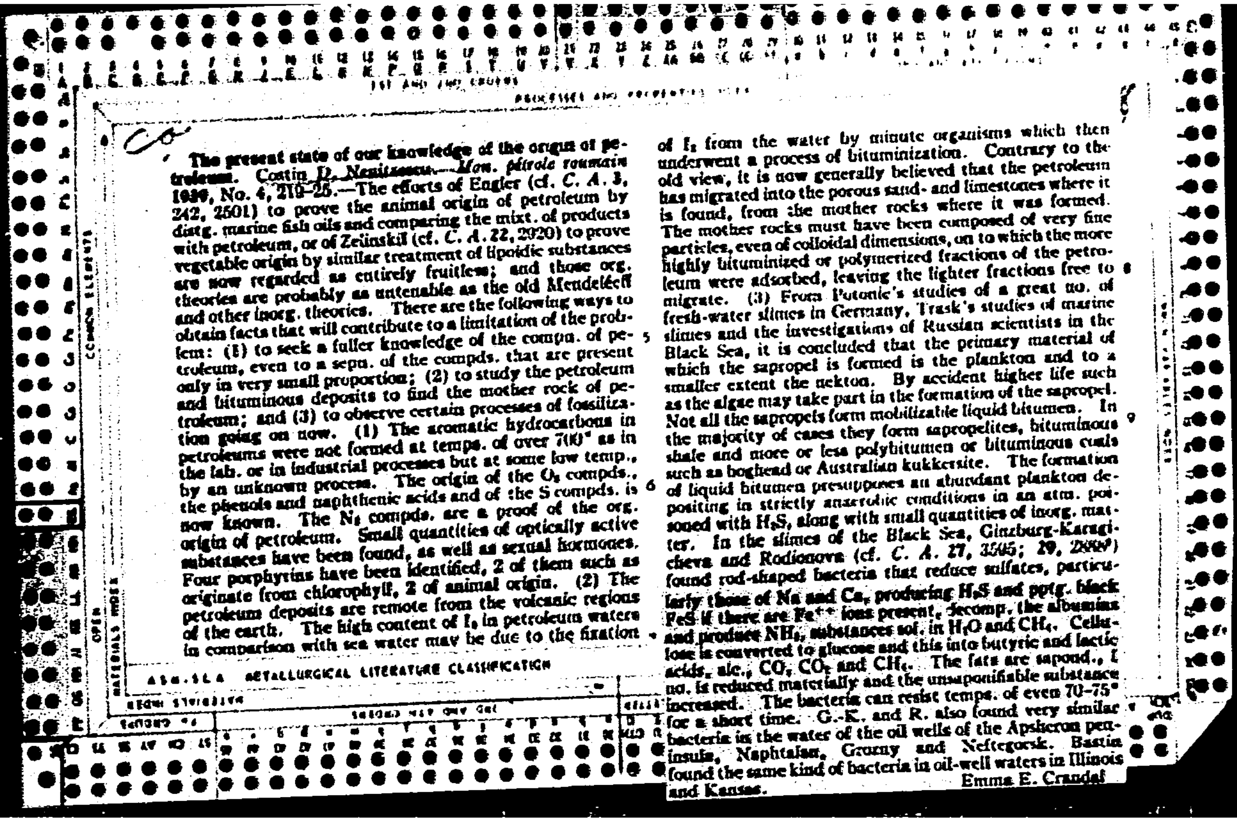
ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION



10

Introduction of arsenic into the aromatic nucleus by means of mercury compounds. Costin D. Nenitzescu, Dumitru A. Isacescu and Carol Gruescu. *Bul. Soc. Chim. Romania* **20A**, 135-8(1938).—A convenient method has been developed for the prepn. of Ph_2AsCl (I) by the action of $AsCl_3$ on PhHgCl and applied also to the prepn. of an arsphenamine intermediate, $4,3\text{-}HO(O_2N)C_6H_3AsO_3H_2$ (II). Thus, heating 2 mols. PhHgCl with 1 mol. $AsCl_3$ for 6 hrs. at 110°, followed by extn. with light benzine, gave 75% of I, m. 44.5°; in another expt. distn. of the product after removal of the benzine gave 8% $PhAsCl_2$ and 61% I. Treating 78 g. $p\text{-}ClC_6H_4HgCl$ with 90 g. $AsCl_3$ for 8 hrs. at 110°, followed by extn. with light benzine and distn. in vacuo, gave 20 g. $p\text{-}ClC_6H_4AsCl_2$, b_{15} 142.5°. Treating 40 g. of the latter in 250 cc. H_2O with Cl to a complete soln., followed by concg. to 180 gave 63% of cryst. $p\text{-}ClC_6H_4AsO_3H_2$, while further concg. gave an addnl. 20%. Treating 7 g. of the arsonic acid in 27 cc. concd. H_2SO_4 with 3.5 g. $NaNO_3$ for 3 hrs. on a water bath and then, upon cooling, with 50 g. ice and 25 cc. H_2O yielded $4,3\text{-}Cl(O_2N)C_6H_3AsO_3H_2$ (cf. Karmer-czak, C. A. **33**, 628), the latter (6 g.) was hydrolyzed with 25 g. 40% KOH for 10 hrs. on a water bath, treated with 12 cc. 27% HCl, decolorized with charcoal, and made acid to Congo test with HCl, which gave 65% of II.

John F. Lontz



Co

The present state of our knowledge of the origin of petroleum. Costin D. Nenitzescu.—*Mon. pétrole roumain* 1939, No. 4, 219-25.—The efforts of Engler (cf. *C. A.* 3, 242, 2501) to prove the animal origin of petroleum by distg. marine fish oils and comparing the mixt. of products with petroleum, or of Zelinskii (cf. *C. A.* 22, 2920) to prove vegetable origin by similar treatment of lipoidic substances are now regarded as entirely fruitless; and those org. theories are probably as untenable as the old Mendeléeff and other inorg. theories. There are the following ways to obtain facts that will contribute to a limitation of the problem: (1) to seek a fuller knowledge of the compn. of pe- 5 troleum, even to a sepn. of the compds. that are present only in very small proportion; (2) to study the petroleum and bituminous deposits to find the mother rock of petroleum; and (3) to observe certain processes of fossilization going on now. (1) The aromatic hydrocarbons in petroleums were not formed at temps. of over 700° as in the lab. or in industrial processes but at some low temp., by an unknown process. The origin of the O_2 compds., 6 the phenols and naphthenic acids and of the S compds. is now known. The N_2 compds. are a proof of the org. origin of petroleum. Small quantities of optically active substances have been found, as well as sexual hormones. Four porphyrins have been identified, 2 of them such as originate from chlorophyll, 2 of animal origin. (2) The petroleum deposits are remote from the volcanic regions of the earth. The high content of I_2 in petroleum waters in comparison with sea water may be due to the fixation of I_2 from the water by minute organisms which then underwent a process of bituminization. Contrary to the old view, it is now generally believed that the petroleum has migrated into the porous sand- and limestones where it is found, from the mother rocks where it was formed. The mother rocks must have been composed of very fine particles, even of colloidal dimensions, on to which the more highly bituminized or polymerized fractions of the petro- 8 leum were adsorbed, leaving the lighter fractions free to migrate. (3) From Potonie's studies of a great no. of fresh-water slimes in Germany, Trask's studies of marine slimes and the investigations of Russian scientists in the Black Sea, it is concluded that the primary material of which the sapropel is formed is the plankton and to a smaller extent the nekton. By accident higher life such as the algae may take part in the formation of the sapropel. Not all the sapropels form mobilizable liquid bitumen. In 9 the majority of cases they form sapropelites, bituminous shale and more or less polybitumen or bituminous coals such as boghead or Australian kukkersite. The formation of liquid bitumen presupposes an abundant plankton depositing in strictly anaerobic conditions in an atm. poisoned with H_2S, along with small quantities of inorg. matter. In the slimes of the Black Sea, Ginzburg-Karagicheva and Rodionova (cf. *C. A.* 27, 3505; 29, 2889) found rod-shaped bacteria that reduce sulfates, particularly those of Na and Ca, producing H_2S and pptg. black FeS if there are Fe^{++} ions present, decomp. the albumins and produce NH_3, substances sol. in H_2O and CH_4. Cellulose is converted to glucose and this into butyric and lactic acids, alc., CO, CO_2 and CH_4. The fats are sapond., I no. is reduced materially and the unsaponifiable substance increased. The bacteria can resist temps. of even 70-75° for a short time. G.-K. and R. also found very similar bacteria in the water of the oil wells of the Apsheron peninsula, Naphtalan, Grozny and Neftegorsk. Bastin found the same kind of bacteria in oil-well waters in Illinois and Kansas.

Emma E. Crandal

PROCESSES AND PROPERTIES INDEX

10

CA

Sweet-tasting compounds of the m-nitroaniline series. Costin D. Nenitzescu and Constantin Bucur. *Bul. Inst. Natl. Cercetări Tehnol.* 3, 15-19(1948)(in French); cf. C.A. 36, 6003.—The reduction of a 1-alkoxy-2,4-dinitrobenzene (I) with Na_2S yields 1-alkoxy-2-nitro-4-aminobenzene (II) and 1-alkoxy-2-amino-4-nitrobenzene (III), sepd. by fractional pptn. of the HCl salts from C_6H_6, the II being more basic than the III. Where alkoxy = PrO, II m. 20° (yield 32%); III m. 48.5° (50%), and 17% I is recovered. Where alkoxy = EtO, III m. 96°.

K. G. Stone

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

10

CA

Polymerization of 1,2-dihydronaphthalene and the dehydrogenating condensation of 1,2,3,4-tetrahydronaphthalene. Costin D. Nenitzescu and Margaret Avram (Scoala Politehnica, Bucharest, Rumania). *J. Am. Chem. Soc.* 72, 3486-90(1950). $PhCH_2CH_2MgBr$ (15 g. bromide) in 120 cc. ether, treated with 8 g. 1-acenaphthenone in 20 cc. C_6H_6 and 80 cc. ether, heated 1 hr., and decompd. with NH_4Cl gives 40% of the crude tertiary alc.; heated 1 hr. at 140° with 30 g. P_2O_5, it yields 44% *6b,7,8,12b-tetrahydrobenzo[j]fluoranthene*, (C.A. numbering), m. 163°; heating 1 hr. at 330° (N atm.) with 27% Pd-C gives quantitatively *benzo[j]fluoranthene* (I), m. 165°, identical with the "bisnaphthylene" of von Braun and Kirschbaum (*C.A.* 15, 2866). $1-C_{10}H_7MgBr$ (15 g. $1-C_{10}H_7Br$) in 120 cc. ether, treated dropwise with 8.8 g. 3,4-dihydro-1(2*H*)-naphthalenone in 70 cc. ether, gives 9 g. crude tertiary alc.; heated 1 hr. at 140° with 15 g. P_2O_5, it gives 3 g. of an oil, b_{10} 200-40°; dehydrogenation with Pd-C gives I; the tertiary alc. distd. *in vacuo* gives 20% *3,4-dihydro-1,1'-binaphthyl*, m. 120.5°. The Mg deriv. from 1,2,3,4-tetrahydro-5-iodonaphthalene in 100 cc. ether treated in the cold with 5 g. 3,4-dihydro-1(2*H*)-naphthalenone, the tertiary alc. heated 1 hr. at 120-5° with 15 g. P_2O_5, distd. *in vacuo*, and the fraction b_7 220-40° dehydrogenated with Pd-C, gives I; partial dehydrogenation occurs during the heating with P_2O_5. Tetrahydronaphthalene (15 g.) and 5 g. 1,2-dihydronaphthalene, heated 1 hr. at 140°, give 2 g. crude hydrocarbon, $C_{20}H_{20}$ (II), b_{10} 225-6°, m. 150.5 (Dansi and Ferri, *C.A.* 37, 113°); dehydrogenation gives I. I (5 g.) in 200 cc. refluxing iso-AmOH with 30 g. Na gives II. The Grignard reagent from 2-bromo-1,2,3,4-tetrahydronaphthalene (from 20 g. tetrahydro-2-naphthol), with 8 g. 3,4-dihydro-1(2*H*)-naphthalenone in 80 cc. ether gives 10 g. of a product yielding with 15 g. P_2O_5 at 130-40° 4-5 g. of crude II, which is therefore *4,5,6,6a,6b,7,8,12b-octahydrobenzo[j]fluoranthene*. The reaction product from the Mg deriv. of 1-bromotetrahydronaphthalene and 3,4-dihydro-1(2*H*)-naphthalenone, treated with P_2O_5, gives an oil, b_9 203-4°, which deposits a small quantity of *1,1',2,2',3,3',4,4'-octahydro-1,1'-binaphthyl*, m. 83°; the oil on dehydrogenation gives I. The hydrocarbon $C_{20}H_{20}$ (m. 93°) of v. B. and K. (obtained by polymerization of 1,2-dihydronaphthalene with H_2SO_4) is probably 1,2,3,6b,7,8,12b,12c-octahydrobenzo[*j*]fluoranthene.

C. I. West

MENITESCU, C.

"Achievements of Soviet chemistry in support of the industrial tasks of the fifth Soviet Five-Year Plan", p. 5 (Analele Romano-Sovietice. Seria Chimie, Series a III-a, v. 5, no. 2, Apr/June 1953, Bucuresti)

East European Vol. 2, No 9
SO: Monthly List of Accessions, /Library of Congress, September 1953, Uncl.

NENITESCU, C.D.

G

Country :Rumania
Category :Organic Chemistry. Synthetic Organic Chemistry.

Abs. Jour. :Ref Zhur-Khimiya, No.12, 1959, No.42374

Author :Nenitescu Costin, D., Avram Margareta, Maxim, N.
Institut. :Not given
Title :Bromine Derivatives of 2-Phthalimidopropane

Orig Pub. :Studii si cercetári chim., 1953, 6, No. 2, 239-245

Abstract :The Bromination of $RC(CH_3)=CH_2$ (I) (both here and below $R=o-C_6H_4(CO)_2N$) with N-bromosuccinimide (II) produces $RC(=CH_2)CH_2Br$ (III) which proved to be identical with one of the isomeric compounds previously obtained (Gabriel S., Ber., 1911, 44, 3035) by heating $RCBr(CH_3)CH_2Br$, synthesized by the action of Br_2 on I, above its melting point. On the basis of this, the formula $RC(CH_3)=CHBr$ should be written for another isomer having a melting point of 150-151°.

Card: 1/2

G

Country : Rumania
Category : Organic Chemistry. Synthetic Organic Chemistry

Abs. Jour. : Ref Zhur-Khimiya, No.12, 19[illegible], No.42[illegible]

Author :
Institut. :
Title :

Orig. Pub. :

Abstract : Substance (III) is hydrogenated (PtO_2, 20° in ethyl acetate). This results in the formation of $RCH(CH_3)_2$ having a melting point of 83°. The addition of Br to III produces $RCBr\cdot(CH_2Br)_2$ (IV) which upon being heated is converted into $RC(CH_2Br)=CHBr$ (V). In the presence of CH_3OH or of alcohol, IV is transformed into $RC(CH_2Br)_2OR'$ where $R'=CH_3$ (VI) or C_2H_5 (VII). The synthesis of I is described according to the following arrangement: $KCN+NH_4Cl+$

Card: 2/7

G

Country :Rumania
Category :Organic Chemistry. Synthetic Organic Chemistry

Abs. Jour. :Ref Zhur-Khimiya, No.12, 1959, No.42374

Author :
Institut. :
Title :

Orig. Pub. :

Abstract :acetone→$(CH_3)_2C(NH_2 \cdot HCl)$ COOH (VIII)→RC·$(CH_3)_2$COOH (IX)→RC$(CH_3)_2$COCl (X)→I. 0.09 mole of II and 0.1 g of $(C_6H_5CO)_2O_2$ are added to 0.1 mole of I in 200 ml. of CCl_4. The mixture is boiled for 48 hours; 62.5% III is then separated from the filtrate; the melting point is 98° (from ligroin or from CH_3OH). 6.4 g of Br_2 in 20 ml. of CCl_4 are added drop by drop to 0.04 mole of III in 50 ml. of CCl_4; the solvent is removed at 10-20°/10 mm, and IV is separated; the melting point is 101° (from

Card: 3/7

G

Country : Rumania
Category : Organic Chemistry. Synthetic Organic Chemistry

Abs. Jour. : Ref Zhur-Khimiya, No.12, 1959, No.42374

Author :
Institut. :
Title :

Orig. Pub. :

Abstract : ether). Unpurified IV (from 0.02 moles of III) is heated in vacuum for 1 hour at 105-110°; the melt is dissolved in hot CH_3OH and a 65% yield of V is separated; the melting point is 172°. CH_3OH is added to unpurified IV (from 0.04 moles of III); the mixture is boiled until the precipitation of HBr stops; a 59% yield of VI is separated; its melting point is 120-121° (from CH_3OH). VII is obtained in a similar manner by the action of alcohol; the melting

Card: [illegible]/7

G

Country : Rumania
Category : Organic Chemistry. Synthetic Organic Chemistry

Abs. Jour. : Ref Zhur-Khimiya, No.12, 1959, No.42374

Author :
Institut. :
Title :

Orig Pub. :

Abstract : point is 135° (from alcohol). 4 moles of KCN, 4 moles of NH_4Cl, 1 liter of water and 4 moles of acetone are heated for 6 hours in an autoclave at 50-60°; the whole is cooled with water containing ice; 3 liters of concentrated HCl is added and in about 12 hours the mixture is boiled with charcoal for 2 hours; the filtrate is vacuum distilled until dry; the residue is extracted with absolute alcohol; the extract is boiled down to 300 ml, and a 72% yield of VIII

G

Country : Rumania
Category : Organic Chemistry. Synthetic Organic Chemistry

Abs. Jour. : Ref Zhur-Khimiya, No.12, 1959, No.42374

Author :
Institut. :
Title :

Orig. Pub. :

Abstract : is separated. 1 mol of VIII is added to a solution of 1 mole of phthalic anhydride (or 166 g of o-$C_6H_4(COOH)_2$) and 2 moles of KOH in 400 ml of water; the mixture is heated, while evaporating the water, to 180-190°. This temperature is maintained for [illegible]0 minutes; upon cooling the mass is pulverized into powder which is macerated with 10% $NaHCO_3$; the filtrate is acidified with concentrated HCl, and a 52% yield of IX is separated off; the melting point is

Card: 6/7

G

Country :Rumania
Category :Organic Chemistry. Synthetic Organic Chemistry

Abs. Jour. :Ref Zhur-Khimiya, No.12, 1959, No.42374

Author :
Institut. :
Title :

Orig Pub. :

Abstract :1540 (from benzoyl). 0.36 moles of IX and 0.66 moles of $SOCl_2$ are heated at 50° for 2 hours; the excess $SOCl_2$ is distilled off in vacuum, and the unpurified X is separated; its melting point is 82°. The CO and HCl are separated by heating the unpurified X to 180-190°; the residue is distilled at 170-180°/10 mm; I is then obtained, its yield is 47%, melting point 105° (from CCl_4).
-- V. Skorodumov

NENITESCU, Costin D.

"Acylamino-methylation, a General Method of Substitution of the Aromatic Ring."Revue de Chemie, Vol. 2, 1954, Bucharest.

CZECH

Rumanian chemistry yesterday and to-day. C. D. Nenitescu. *Chem. Průmysl* 4(29), 365-8(1954). The development of the Rumanian chemical industry from its beginnings is briefly reviewed. L. A. Helwich

Nenitzescu, C. D.

The mechanism of alkylation of aromatic nuclei. I. Kinetic studies of the condensation of benzene hydrocar-

NENITESCU, C.D.

RUMANIA/Chemical Products and Their Application. Synthetic Polymers. Plastics. H-29

Abs Jour: Ref. Zhur-Khimiya, No 11, 1958, 38029.

Author : Nenitescu C D, Costescu Dan C
Inst : Not given.
Title : The possibilities of Substituting Synthetic Polymers for Nonferrous Metals

Orig Pub: An Acad RPR, 1955, 3, Anexa, 129-142.

Abstract: A review of the structure and properties of various plastics. It formulates problems that are before the Academy of Sciences RPR in connection with the question of substituting plastics for nonferrous metals.

Card : 1/1

NENITESCU, K.D., professor.

Development of chemistry in Rumania. Priroda 44 no.12:65-67
D '55. (MIRA 9:1)
(Rumania--Chemistry)

Nenitescu, C. I.

RUMANIA/Organic Chemistry. Theoretical and General Questions of Organic Chemistry. E-1

Abs Jour : Ref Zhur - Khimiya, No. 8, 1957, 26670 K.

Author : Nenitescu, C.I.
Inst : Bucharest Polytechnical Institute.
Title : Organic Chemistry. Volume I.

Orig Pub : 3rd edition; Ministerul Invătămîntului. Inst. politehn., Bucureşti, 1956, 588 p., ill., 12 lei. - Litogr.

Abstract : No abstract.

Card 1/1

NENITESCU C.

NENITZESCU, C. ; IOAN, V.

About the ploymeric pyrroles. In German. p. 55.
(REVUE DE CHIMIE. RUMANIA. Vol 1, no. 1, 1956)

SO: Monthly List of East European Accessions (EEAL) LC, Vol. 6, no. 7, July 1957. Uncl.

NENITZESCU, C. ; AVRAM, M. ; SLIAM, E.

Reduction of the 5-nitrouracils with sodium dithionite. In German. p. 73.
(REVUE DE CHIMIE. RUMANIA. Vol 1, no. 1, 1956.)

SO: Monthly List of East European Accessions (EEAL) LC, Vol. 6, no. 7, July 1957. Uncl.

NENITZESCU, C. ; BUCUR, C.

About a new preparation and some derivatives of the 5 - nitrofurfural. In German. p. 155.
(REVUE DE CHIMIE. RUMANIA. Vol. 1, no. 1, 1956.)

SO: Monthly List of East European Accessions (EEAL) LC, Vol. 6, no. 7, July 1957. Uncl.

NENITESCU, ~~C.~~

NENITESCU, C.; AVRAM, M.; SLIAM, E. Reduction of 5-nitrouracil by sodium dithionite.

Vol. 4, No. 1/2, Jan/June 1956 Bucuresti, Rumania

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 5, No. 10, Oct. 1956

NENITESCU, C.

About a relation between the melting point and the number of carbon atoms in a series of superior normal paraffins. p. 65.

STUDII SI CERCETARI DE CHIMIE

Vol. 4, No. 1/2, Jan./June 1956

Rumania

SOURCE: EEAL, Vol. 5, No. 10 Oct. 1956

NENITESCU

G-2

RUMANIA/Organic Chemistry. Synthetic Organic Chemistry.

Abs Jour: Ref. Zhur.-Khimiya, No II, 1958, 36203.

Author : Nenitescu, Necsoiu, Zelman.
Inst : ~~Not given.~~
Title : Synthesis of Pyrrole Ring by Condensation of α-oxyketones with β-Aminocrotonic Ester.

Orig Pub: Comun. Acad. RPR, 1957, 7, No 4, 421-426.

Abstract: A number of derivatives of pyrrole with a general formula $\overline{C(COOC_2H_5)=C(CH_3)\ NHC(R)=CH}$ were synthesized by condensation of α-oxitones with ethyl ester of β-aminocrotonic acid (I). (In the above formula II, when R=CH_3, III R=C_2H_5, IV R=C_3H_7, V R=C_6H_5, VI R=α-C_4H_3O). From acetone, cyclopentamolone-2, and cyclohexanolone-2 were obtained respectively 2, 5-dimethyl-3-carbethoxypyrrol (VII), 2-methyl-3-carbethoxy-4,5-cyclohexanepyrrol (IX). In the

Card : 1/3

yield of VII was 54%. Analogically acetoin, propyoin, butyroin, benzoin, and furoin reacted with I while being heated on a steam bath for 0.3-2 hours (for the obtainment of IX -I hour at 120°C) in solutions with either CH_3COOH or with CH_3COOH + CH OH (1/1 ration) yielding

Card : 2/3

10

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

Abs Jour: Ref. Zhur.-Khimiya, No II, 1958, 36203.

II - VI, VIII, IX. Given below are yields in % and melting points in °C: II 85.3, 105; III 92, 109; IV 94, 92 (from CH_3OH); V 89, 202; VI 74.5, 115 (from CH OH); VIII 26, 156 (from CH_3OH); IX 61, 132 (from 80% CH_3OH). An attempt to apply the same condensation method to α-oxaldehyde with I, α-glucose and α-fructose failed.

Card : 3/3

NENITESCU, C., AND OTHERS

The polymerization of ethylene at normal pressure. Note 2.

P. 395 (REVISTA DE CHIMIE) (Bucuresti, Rumania) Vol. 8 No. 6. June 1957

SO: Monthly Index of East European Accessions (EEAI) LC Vol. 7, No. 5. 1958

4

Cyclobutane series. I. 1,3-Disubstituted cyclobutane derivatives. Margarete Avram, Costin D. Nenitzescu, and Maria Maxim (Akad. R.V.R., Bucharest, Romania). *Chem. Ber.* 90, 1424–32(1957); cf. *C.A.* 54, 4512*b*.—The synthesis of 1,3-diaminocyclobutane (I) as well as several intermediate products and derivs. was described. Epibromohydrin (246 g.) and 290 g. $PhCH_2Br$ heated 8 hrs. at 155–60° (internal temp.) with 0.2 g. $HgCl_2$ and the product distd. gave 343 g. crude $PhCH_2OCH(CH_2Br)_2$ (II), b_3 148–55°; the forerun (b_{14} to 150°) heated again with $HgCl_2$ and the product distd. gave 64 g. II, b_3 148–55°; on repeated distn. II b_3 148°. Epichlorohydrin (340 g.) and 615 g. $PhCH_2Br$ treated with 0.5 g. $HgCl_2$, heated slowly (during 5 hrs.) to 155°, kept 6 hrs. at 155–60°, and distd. gave 608 g. $PhCH_2OCH(CH_2Br)CH_2Cl$ (III), b_6 146–50°; from the forerun an addnl. 102 g. III was obtained; redistd. III b_6 146°. Na (23 g.) was dissolved in 400 cc. EtOH, half the soln. and 88 g. $CH_2(CO_2Et)_2$, 400 cc. dry C_6H_6, and 154 g. crude II (or 132 g. crude III) heated 1.5 hrs. at 170° (bath temp.) in a 1.5 l. autoclave (pressure 6 atm.), cooled (neutral soln.), 400 cc. dry C_6H_6 and the remaining NaOEt soln. added, heated 6 hrs. (with III 9 hrs.) at 170°, the cooled neutral soln. filtered from KBr (or KCl), and the filtrate concd. on a H_2O bath; the KBr was dissolved in H_2O, the soln. extd. with Et_2O, the ext. concd., the residue combined with the main fraction, and repeatedly distd. to give 65 g. crude $CH_2.CH(OCH_2Ph).CH_2.C(CO_2Et)_2$ (IV), b_3 178–83°, on redistn. b_3 178°, d_{25} 1.0983, n_D^{25} 1.4819. Crude IV (190 g.) and 170 g. KOH in 420 cc. EtOH and 100 cc. H_2O refluxed 2 hrs. on a H_2O bath, evapd. *in vacuo* almost to dryness, the residue dissolved in 400 cc. H_2O, the soln. extd. with Et_2O, acidified with concd. HCl (soln. V), and the ppt. filtered off gave $CH_2.CH(OCH_2Ph).CH_2.C(CO_2H)_2$, m. 158° ($H_2O$). Soln. V prepd. as above extd. with 5 100-cc. portions Et_2O, the ext. dried, evapd., the residue heated at 10 mm. until CO_2 evolution ceased, and distd. gave 98 g. $CH_2.CH(OCH_2Ph).CH_2.CHCO_2H$ (VI), b_6 194°, d_{25} 1.13907. VI (2 g.) and 1 g. urea heated 3 hrs. at 160–5° gave the amide, m. 120° (H_2O). VI (93 g.), 50 cc. abs. EtOH, 80 cc. C_6H_6, and 2 drops H_2SO_4 refluxed 8 hrs. (H_2O separator used) gave 100 g. VI Et ester (VII), b_6 169°, b_{18} 175°, d_{25} 1.0632, n_D^{25} 1.5010, *MR* 65.00. VII (117 g.) in 500 cc. EtOH mixed with 0.5 g. Pd black, shaken with H at atm. pressure, after absorption of the calcd. amt. H (5–8 hrs.) the catalyst filtered off, the filtrate concd., and the residue distd. gave 35 g. $CH_2.CH(OH).CH_2.CHCO_2Et$ (VIII), b_{16} 120°, d_{25} 1.0727, n_D^{25} 1.4504, *MR* 36.13. VIII (30.4 g.) and 35.2 g. $PhSO_2Cl$ cooled in ice H_2O, treated portionwise with 20 cc. dry C_5H_5N, the mixt. kept 20 hrs. in an ice box, treated with 30 cc. H_2O, extd. with Et_2O, the Et_2O ext. washed successively with H_2O, dil. HCl, aq. Na_2CO_3, and H_2O, dried, and evapd. *in vacuo* (60° bath temp.) gave 65 g. $PhSO_2$ ester (IX) of VIII, purity 99.7%. IX (66 g.) dild. with 55 cc. EtOH, treated with 24 g. NaN_3 in 25 cc. H_2O, the mixt. boiled 10 hrs., cooled, the sepd. $PhSO_2Na$ dissolved by adding H_2O, the soln. extd. with Et_2O, the ext. washed with H_2O, and dried gave 27 g. $CH_2.CHN_3.CH_2.CHCO_2Et$ (X), b_6 94°, d_{25} 1.0902, n_D^{25} 1.4607, *MR* 42.54. X (25.5 g.) and 15 cc. $N_2H_4.H_2O$ (XI) heated 30 min. on a boiling H_2O bath, the excess XI evapd. *in vacuo* on the H_2O bath, and the residue cooled gave 23 g. hydrazide (XII), m. 76° (C_6H_6-ligroine). XII (20 g.) dissolved in 270 cc. cold (0°) *N* HCl, mixed with 150 cc. Et_2O, the mixt. treated dropwise at 0–5° with aq. $NaNO_2$

Avram, M.; [illegible]; [illegible] 4

(10 g. $NaNO_2$ in 50 cc. H_2O) until free HNO_2 formed (detectable with KI-starch paper) the aq. phase sepd., extd. with 2 50-cc. portions Et_2O, the ext. dried by stirring at 0° with $CaCl_2$, filtered, the Et_2O filtrate mixed with 50 cc. $PhCH_2OH$, the Et_2O slowly (2 hrs.) distd. (N evolution began at 20°), after the removal of the Et_2O the mixt. heated 1 hr. at 80° (N evolution ceased), and the excess $PhCH_2OH$ distd. at 120-30° (oil bath temp.)/5 mm. gave 24 g. $\overline{CH_2.CHN_2.CH_2.CH}NHCO_2CH_2Ph$ (XIII), m. 66° (ligroine). XIII (12.3 g.) in 100 cc. EtOH and 0.2 g. Pd black shaken at 30-5° in an H atm. [the H often removed until it contained no more CO_2 (6 hrs.)] and the mixt. worked up as usual gave 2.3 g. I, b$_{14}$ 90°; dipicrate m. above 250° (EtOH); hexachloroplatinate, crystals from H_2O. I (1.72 g.) heated 10 hrs. at 100° with 18.8 g. 95% HCO_2H and 10 g. 35% aq. CH_2O, the mixt. made alk. with 40% aq. NaOH, extd. with Et_2O in a Kutscher-Steudel app., and the product distd. gave 0.4 g. forerun, b$_{14}$ to 70°, and 1.8 g. *N,N,N',N'*-tetra-Me deriv. (XIV) of I, b$_{14}$ 85° (dipicrate m. above 250°). XIV (3.5 g.) in 10 cc. abs. Et_2O treated with 7.5 cc. MeI and after 6 hrs. the product completely pptd. with Et_2O gave XIV di-MeI salt, m. 240-60° (MeOH-H_2O); dipicrate m. above 250°. VII (16 g.) and 20 cc. 80% XI heated 10 min. on the H_2O bath until homogenized, allowed to stand 2 hrs. at room temp., most of the liquid removed *in vacuo*, the residue treated with 10 cc. XI, and allowed to crystallize (12 hrs.) gave 13 g. hydrazide (XV) of VI, m. 100° (EtOH). XV (11 g.) dissolved in 100 cc. *N* HCl, treated dropwise with stirring with a concd. soln. of 3.5 g. $NaNO_2$ at 0-5°, the oily azide extd. with 150 cc. cold (0°) Et_2O, the ext. dried at 0° (0.25 hr.), treated with 150 cc. abs. EtOH, the Et_2O evapd. with a downward condenser (N evolution started at 50-60°), finally 50 cc. EtOH added, the soln. refluxed 1 hr., and the EtOH removed *in vacuo* gave 10 g. $\overline{CH_2.CH(OCH_2Ph).CH_2.CH}NHCO_2Et$ (XVI), m. 68° (EtOH or ligroine). XVI (9 g.) and 25 g. KOH in 70 cc. MeOH refluxed 1 hr., the MeOH distd., the residue mixed with H_2O, the mixt. extd. with Et_2O, the ext. washed with a little H_2O, dried, and evapd. gave 6 g. $\overline{CH_2.CH(OCH_2Ph).CH_2.CH}NH_2$ (XVII), b$_4$ 118°, d$_{25}$ 1.0315, n_D^{25} 1.52935, *MR* 53.0. XVII (8 g.) and 15 cc. Ac_2O boiled a short time and cooled gave the Ac deriv., m. 95° (ligroine); the Ac_2O filtrate concd. and the residue distd. gave an unidentified compd., b$_7$ 210°, m. 75° (ligroine). Hydrogenation of XVII with Pd black failed because of balling of the catalyst. To a complex of 59 g. CrO_3 in 590 cc. C_5H_5N prepd. at 0° was added portionwise 28 g. VIII in 280 cc. C_5H_5N keeping the temp. below 20°, the mixt. kept 12 hrs. at room temp. and 1 hr. at 45°, dild. with ice H_2O, and extd. with 1:1 Et_2O-C_6H_6 gave 18.1 g. $\overline{CH_2.CO.CH_2.CH}CO_2Et$, b$_2$ 90°; semicarbazone m. 205° (H_2O); 84% oxime (XVIII), b$_4$ 145-6°. XVIII (5 g.) in 500 cc. EtOH hydrogenated with 0.5 g. PtO_2 at 30-5° under normal pressure gave 1.7 g. $\overline{CH_2.CH(NH_2).CH_2.CH}CO_2Et$ (XIX), b$_{12}$ 88°, and 2.4 g. fraction (apparently secondary amine), b$_7$ 180-5°, not further investigated. XIX gave an *N*-Bz deriv. (XX), m. 104.5-5.0° (ligroine), an *N*-phthalyl deriv., m. 60° (ligroine), and was converted into 3-phthalimido-1-cyclobutanecarboxylic acid, m. 178-80° (H_2O). XX treated with XI and the resulting

Alleam, Margareta; [illegible]; Costin D; 4
and Maxim, [illegible]

hydrazide subjected to a Curtius degradation gave $\overline{CH_2.CH(NHBz).CH_2.C}HNHCO_2Et$, m. 185° (aq. EtOH). IV reduced like VII gave 85% $\overline{CH_2.CH(OH).CH_2.C}(CO_2Et)_2$ (XXI), b_2 120-2°, d_{20} 1.1108, n_D^{20} 1.4488, *MR* 52.17. XXI oxidized like VIII gave 75% oxo diester, b_1 112-13°; semicarbazone m. 176°; 78% oxime (XXII), b_1 127°, d_{20} 1.1583, n_D^{20} 1.46855, *MR* 55.03. XXII (12 g.) catalytically reduced like XVIII gave 4 g. amino diester, b_2 98°, and 6 g. compd., b_2 195°, apparently the secondary amine. IX (10 g.) in 10 cc. EtOH treated with 10 g. KI in 5 cc. H_2O, boiled 10 hrs. on a H_2O bath, and worked up gave 5 g. $\overline{CH_2.CHI.CH_2.C}HCO_2Et$ (XXIII), b_9 113° (decompn.). XXIII (5 g.) in 100 cc. 80% EtOH treated portionwise during 12 hrs. with 150 g. 3% Na-Hg with stirring, the supernatant liquid sepd. from Hg, boiled 6 hrs., acidified, and extd. with Et_2O gave 1.5 g. $\overline{CH_2.CH_2.CH_2.C}HCO_2H$, b_{14} 110°, b. 194°; *S*-benzylthiuronium salt m. 176° (EtOH). A more convenient method of prepg. I from XII was as follows. XII (31 g.) was converted, like in the prepn. of XIII but replacing the $PhCH_2OH$ by 100 cc. abs. EtOH, into 32 g. crude $\overline{CH_2.CHN_3.CH_2.C}HNHCO_2Et$ (XXIV). Crude XXIV (32 g.) subjected to a Curtius degradation gave 13 g. $\overline{CH_2.CHN_3.CH_2.C}HNH_2$, b_{20} 65°, hydrogenated with Pd black to 60% I, b. 155° [1,3-di-Ac deriv. m. 218° (EtOH)].

Ek

William

NEMITZESCU, C-D.

4E3b
4E3d
4E2c(j)

/ Investigations in the cyclobutene series. II. *Cyclobutadiene?* *Margarete Avram, Costin D. Nenitzescu,* and *Elise Marica* (Acad. R. P. R. Bucarest, Romania). *Chem. Ber.* 90, 1857-68(1957); cf. *C.A.* 51, 16993*f*.—Hofmann degradation of 1,2-bis(dimethylamino)cyclobutane as well as thermal fission of Reppe adducts from cyclooctatetraene and several dienophiles result only in formation of butadiene (I) instead of the expected cyclobutadiene. A soln. of 1.8 g. 1,2-bis(dimethylamino)cyclobutane-2MeI (II) in 20 ml. MeOH is shaken 1 hr. with Ag_2O (from 5 g. $AgNO_3$ and 10% aq. NaOH). After filtration and washing of the ppt. with MeOH, the filtrate is evapd. at 20-35° *in vacuo*. From a bath temp. of 120° up to 200°, decompn. with formation of 20 ml. gas, which is caught over 50% KOH, takes place; the gas has a C-H ratio of 1:1.5. II (4.26 g.) is heated with 5 g. KOH in 5 ml. water for 1.5 hrs.; the resulting gas is led into a soln. of Br in CH_2Cl_2. After evapn. a small quantity 1,2,3,4-tetrabromobutane, m. 117°, is left. II (4.26 g.) after treatment according to the first procedure, on distn. of the filtrate, gives 2 ml. MeOH, which on diln. with water yields an oil. After extn. with ether, drying, and evapg., the yellow oil (50 mg.) is mixed with 50 mg. maleic anhydride to give III, m. 97° (dioxane-ether) (Fiaig, *C.A.* 44, 6808*d*). $(MeO_2CCH)_2$ (28 g.) and 23 g. cyclooctatetraene is heated 8 hrs. at a bath temp. of 150-5°. At slightly higher temp. decompn. takes place to give 25 g. IV, b_1 140-150°. On heating 0.1 mole (24 g.) IV 15 min. to about 300°, 350-420 ml. gas is vigorously evolved and proves to be I (15-17% yield). From the residue there is obtained 4-6 g. di-Me 2,6-naphthalenedicarboxylate (V), m. 187°, 6-6.5 g. (30-32) di-Me phthalate, b_1 140-50°, and 6.5 g. VI, m. 120°. At 300° with 30% Pd-C VI gives V. When VII (R = Me), m. 105° (Reppe, *et al.*, *C.A.* 43, 6194*g*), is heated to the b.p., I is obtained in 25% yield, and from the residue 50-54% 1,4-dimethoxynaphthalene, m. 85°, and 1,4-dimethoxyphenanthrene, m. 124.5-125°, are isolated and identified. VII (R = Ac), m. 136-7° (*loc. cit.*), gives on thermal degradation 1,4-diacetoxynaphthalene, m. 123-6°, and 1,4-diacetoxyphenanthrene, m. 137°. Fission of the adduct of cyclooctatetraene and α-naphthoquinone, m. 190-2°, gives I, anthraquinone, and a mixt. of isomeric benzanthraquinones, m. 150° (MeOH) (the mixed m.p. with authentic 1,2-benzanthraquinone, m. 168°, is 155°).

C. R. Freitag

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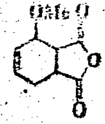
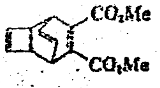


(III)



(IV)

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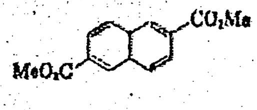
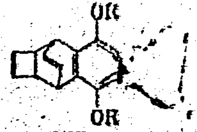


(VI)



(VII)

NENITZESCU, Costin D.

Distr: 4E2c(j)/4E3d

Cyclobutanes. III. Benzocyclobutadiene. Costin D. Nenitzescu, Margareta Avram, and Doina Dinu (Tech. Hochschule, Bucharest, Romania). *Chem. Ber.* 90, 2541-4 (1957); cf. *ibid.* 1857.—Formation of dihydrobenzodiphenylene (I) from 1,2-dibromobenzocyclobutene (II) and Zn (cf. Cava and Napier, *C.A.* 51, 11310g) suggests that *benzocyclobutadiene* (III) is a distinct but short-lived intermediate. The cyclopentadiene (IV) *adduct* of III is now obtained by reaction of II with Li-Hg, then IV. Similar reaction with furan under these conditions gives an amorphous polymer. A soln. of 80 g. o-$C_6H_4(CHBr_2)_2$ in 600 cc. Me_2CO (very dry) is refluxed 52 hrs. with 105 g. NaI in 700 cc. Me_2CO, filtered, the soln. evapd. to 750 cc., decolorized with $Na_2S_2O_3$, and dild. to 1 l. with H_2O, the ppt. sepd., and the soln. extd. with Et_2O gives 50% II, m. 62-3°. A soln. of 3 g. II in 50 cc. abs. Et_2O is shaken 10-12 hrs. with 100 g. 0.5% Li-Hg (cooling at first), sepd., washed with H_2O, and evapd. to give 80% I, m. 74°. A soln. of 15 g. II in 25 cc. abs. Et_2O shaken with 500 g. 0.5% Li-Hg and 19 g. IV gives 52% *1,4-endomethylene-1,4,11,12-tetrahydrodiphenylene* (V), b_7 82-4°, n_D^{20} 1.57817, d_{20} 1.0539. V absorbs 1.2 moles H over Pd. V forms *adducts* with PhN_3 (m. 182°) and CH_2N_2 (m. 70°). The latter heated to the b.p. in a CO_2 stream forms pyrazole. M. A. Simkins

jb

1/1

Distr: 4E2c(j)/4E3d 1

3 May

Brominated derivatives of 2-phthalimidopropane. Costin D. Nenitzescu, Margareta Avram, and Maria M[illegible]. *Acad. rep. populare Romîne, Studii cercetări chim.* 6, 239-45 (1958).—α-Phthalimidoisobutyric acid (I) was prepd. from K H phthalate and $HO_2CCMe_2NH_2.HCl$. From the acid chloride of I 2-phthalimidopropene (II), m. 105° was obtained; on bromination with *N*-bromosuccinimide II yields 1-bromo-2-phthalimidopropene (III), m. 98°. Brominating III gives 1,2,3-tribromo-2-phthalimidopropane (IV), m. 101°. The 2-Br of IV is easily substituted by methoxy or ethoxy groups to yield, resp., 1,8-dibromo-2-methoxy-2-phthalimidopropane, m. 117-18°, and 1,3-dibromo-2-ethoxy-2-phthalimidopropane, m. 135°. J. Segall

JI

1/1

G-2

COUNTRY : Rumania
CATEGORY :

ABS. JOUR. : RZKhim., No. 21 1959, No. 74864

AUTHOR : Nenitescu, C. D., Pogany, I., and Minai, G.
INST. : Rumanian Academy of Sciences
TITLE : Effect of Ring Size on the Reaction of Simple C_5 -C_8 Cycloolefins with Acetyl Chloride in the Presence of Aluminum Chloride

ORIG. PUB. : Studii si Cercetari Chim Acad RPR, v. No 3, 375-389 (1958)

ABSTRACT : The reaction of 0.1 mol CH_3COCl with 0.1 mol cycloolefin in 60 ml isopentane or CS_2 in the presence of 0.22 mol $AlCl_3$ (the $AlCl_3$ is added over 2 hrs at a temperature of from -15 to -10° and the solution is held at that temperature for 7 hrs) gives mixtures of the following products (the starting cycloolefin and the yields of the corresponding β-, γ- or δ-, unsaturated and saturated ketones in isopentane and in CS_2 in % are given): cyclopentene, 6 and 4, 65 and 86,

CARD: 1/6

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COUNTRY : Rumania G-2
CATEGORY :

ABS. JOUR. : RZKhim., No. 21 1959, No. 74864

AUTHOR :
[illegible] :
TITLE :

ORIG. PUB. :

ABSTRACT : 4 and 10. 25 and -; cyclohexene, 47 and 39, 16 and 17.9, 44.28 and -; cycloheptene, 7 and 20. ~ 7 and 10, 4 and 70, 82 and -; cyclooctene, 4 and 5, 1 and 6, 1 and 39, 94 and -. A shrinking of the ring is observed during the condensation of CH_3COCl with cycloheptene and cyclooctene in isopentane. Hydrogenation of the products obtained from the latter reactions over $Pd/CaCO_3$ in alkaline medium gives saturated ketones having the composition $CH_3COC_7H_{13}$ (semicarbazone (SC) mp 170° (from 50% alc and ethyl acetate)) and

CARD: 2/6

COUNTRY : Rumania
CATEGORY :
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ABS. JOUR. : RZKhim., No. 21 1959, No. 74864
AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : $CH_3COC_8H_{15}$ (SC mp 170-172° (from 50% alc)), re-
spectively. These ketones were oxidized with
NaOBr to the corresponding acids from which the
respective amides $C_7H_{13}CONH_2$, mp 176° (from
water), and $C_5H_{15}CONH_2$, mp 191° (from water),
were prepared via the acid chlorides. 38 gms of
a mixture of ketones (mainly methyl-(3-chloro-
cyclopentyl)-ketone (I)), obtained by the reac-
tion of CH_3COCl with cyclopentene and isopentane
followed by the hydrogenation of the reaction

CARD: 3/6

G-2

COUNTRY : Rumania
CATEGORY :

ABS. JOUR. : RZKhim., No. 21 1959, No. 74864

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : products over Pd, was boiled 4 hrs with 50 gms CH_3COOK in 80 gms glacial CH_3COOH yielding 21 gms of crude metnyl-(3-acetoxycylopentyl)-ketone, bp 110-125°/10 mm, which on oxidation with NaOBr gave 22 gms of a mixture consisting of 73.5% 3-hydroxycyclopentane-1-carboxylic acid and 26.5% 3-acetoxycyclopentane-1-carboxylic acid. Further oxidation of the mixture with chromium trioxide yields cyclopentanone-3-carboxylic-1 acid (II) bp 155°/6 mm, mp 60° (from petroleum ether), SC

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CARD: 5/6

COUNTRY : Rumania G-2
CATEGORY :

ABS. JOUR. : RZKhim., No. 21 1959, No. 74864

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : oxidation of III with NaOBr and chromium dioxide gives II, yield 22.5%, DNPH of the ethyl ester of II mp 148° (from alc). The oxidation of III with chromium dioxide gives methyl-(3-ketocyclopentyl)-ketone, SC mp 234°, bis-DNPH mp 239° (from C_5H_5N). The mechanism by which the various ketones are formed during the reaction of CH_3COCl with cycloolefins in the presence of $AlCl_3$ is discussed.

S. Zav'yalov

CARD: 6/6

7

Diels-Alder condensation of 3,4-benzisoxazole with N-phenylmaleimide. C. D. Nenitzescu, Beatricia Cioranescu, and Ludmila Birladeanu. *Comun. acad. rep. populare Romîne* 8, 775-9(1958).—Endo and exo isomers of 1,4-endoxy-1,2,3,4-tetrahydroquinoline-2,3-dicarboxylic *N*-phenylimide are obtained on heating equivalent amts. of 3,4-benzisoxazole and *N*-phenylmaleimide to 125 and 140°, resp. The endo isomer m. 100°, heated to 140° gives the exo isomer, m. 283°. Alk. hydrolysis yields acridinic acid. From these data the structure of 3,4-benzisoxazole is confirmed. J. Segall

2May
4E2c(j)
4

Nenitescu, C.D

RUMANIA / Organic Chemistry--Synthetic organic chemistry. G-2

Abs Jour : Ref Zhur - Khimiya, No 14, 1959, No. 49514

Author : Nenitescu, C. D.; Necsoiu, I.; Zalman, M.
Inst : Rumanian Academy of Sciences
Title : The Condensation of Oxalyl Chloride with Substituted Pyrroles

Orig Pub : Comun Acad RPR, 8, No 7, 659-663 (1958)

Abstract : The gradual addition of a solution of 2,4-dimethylpyrrole in a mixture of petroleum ether and ether (1 : 1) to a small excess of $(COCl_2)_2$ in the same solvent (at temperature of from -25 to -30° under an atmosphere of N_2) results in the formation of a precipitate of 3,5,3',5'-tetramethyl-pyrryl-2-diketone, yield 15.5%, mp 245° (from aqueous alc); the mother

Card 1/2

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RUMANIA / Organic Chemistry--Synthetic organic chemistry. G-2

Abs Jour : Ref Zhur - Khimiya, No 14, 1959, No. 49514

solution on removal of the solvent and hydrolysis of the residue with hot 10% NaOH gives 2,4-dimethylpyrroleglyoxylic-5 acid (yield 65%), mp 167° (decomp). The reaction of $(COCl)_2$ with 2,5-dimethyl-3-carbethoxypyrrole in ether (-10°, followed by refluxing for 2 hrs) gives 2,5,2',5'-tetramethyl-4,4'-dicarbethoxypyrryl-3-diketone, yield 22%, mp 234 - 235° (from aqueous alc), and 2,5-dimethyl-3-carbethoxypyrrolecarboxylic-4 acid, yield 40%, mp 234° (decomp; from aqueous alc); the latter product is converted to 2,5-dimethyl-3-carbethoxypyrrole (mp 118 - 119°) by thermal decarboxylation. -- D. Vitkovskiy

Card 2/2

AUTHORS: Nenitsesku, K. D., Iliye G.Dinulesku SOV/62-58-1o-11/25

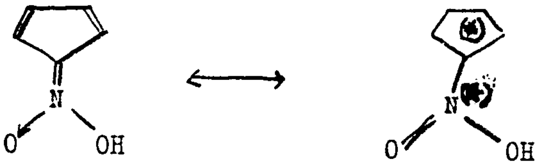
TITLE: The Equilibrium Between the Iso and the Normal Form of Nitrofluorene (Ravnovesiye mezhdu izo- i normal'noy formami 9-nitrofluorena)

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1958, Nr 1o, pp 1228 - 1232 (USSR)

ABSTRACT: In their earlier papers Nenitzescu and **Isacescu** showed (Ref 9) that iso-9-nitrofluorene has a considerable stability. The normal form of this nitro derivative could, however, not be separated. The normal form of 9-nitro fluorene (Ref 1o) proved to be an equimolecular mixture of 1,2-dinitro-1,2-dibiphenyl ethane and fluorenone oxime. All attempts to convert the isoform into the normal form resulted in a mixture of the two substances. The authors of the present paper proceeded from iso-9-nitro fluorene (synthesized by Vislitsenus and Val'dmyuller, Ref 10) as initial substance. They carried out a number of bromometric titrations of the iso-9-nitro fluorene solutions in ethanol, dioxane and benzene. In crystalline

Card 1/3

state the isoform is thermodynamically more stable than the normal form. In solution an equilibrium was attained in which the normal form is the more contained the less polar the solvent is. The normal form of 9-nitro fluorene can not be separated as it is disproportionated by the formation of 1,2-dinitro-1,2-dibiphenyl ethane and fluorenone oxime. This reaction takes place more slowly than the reaction of the formation of the normal form from the iso form. Therefore there are at certain times considerable concentrations of the normal form in the solvent which were found in the chemical way. The great stability of the isoform of 9-nitro fluorene is given by the mesomerism between the cyclopentadiene nucleus and the nitro group:



The Equilibrium Between the Iso and the Normal Form of Nitrofluorene SOV/62-58-10-11/25

There are 2 tables and 16 references.

ASSOCIATION: Otdel khimicheskikh issledovaniy Akademii Rumynskoy Narodnoy Respubliki (Department of Chemical Investigations AS Roumanian People's Republic)

SUBMITTED: May 9, 1958

Card 3/3

: NENITZESCU, COSTIN D.

6
2-may

7 7 - 7

Syntheses of heteroauxin, tryptamine, and serotonin. Costin D. Nenitzescu and Dan Răileanu (Akad. Romanian Peoples Republic, Bucharest, Romania). *Chem. Ber.* 91, 1141-5(1958).—Powd. NaOAc (80 g.) added with stirring to 500 cc. Ac_2O, the mixt. treated rapidly with 100 g. *o*-$HO_2CC_6H_4NAcCH_2CO_2H$, m. 211°, refluxed 5 min. (CO_2 evolution), distd. to remove the excess Ac_2O, the residue heated with stirring with 750 cc. H_2O to boiling, cooled, filtered, the crude 1,3-diacetylindoxyl (about 85 g.) added to 120 g. $Na_2SO_3.7H_2O$ in 1.6 l. H_2O at 70°, the mixt. stirred 1.5 hrs. at 70° and cooled, and the product filtered off yielded 44 g. *1-acetylindoxyl* (I), m. 138°; *oxime*, m. 177° (decompn.) (50% EtOH); *2,4-dinitrophenylhydrazone*, m. 202° (decompn.) (pyridine). NH_4OAc (4 g.) in 75 g. PhOH warmed, treated with 15 g. $NCCH_2CO_2H$, 50 cc. xylene, and 15 g. I, refluxed 5 hrs. with the azeotropic removal of the H_2O, the mixt. steam distd. to remove the xylene, and the steam distn. residue allowed to stand overnight and recrystd. from aq. EtOH gave 10.5 g. *1-acetyl-3-indolylacetonitrile* (II), m. 112° (CCl_4). Crude II (20 g.) and 300 cc. 30% aq. NaOH refluxed 6-7 hrs. and acidified gave 13.5 g. *3-indolylacetic acid* (heteroauxin) (III), m. [illegible]5° (decompn.). II (1.2 g.) extd. into 1.0 g. $LiAlH_4$

stirring at reflux during 2 hrs. with 200 cc. 17% aq. NaOH, refluxed 1 hr., freed of the EtOH, dild. with 600 cc. H_2O, washed with C_6H_6, treated with C, acidified with concd. HCl, and the cryst. ppt. recrystd. at −10° from 200 cc. EtOH and 100 cc. H_2O gave 38 g. *2,5-$Cl(PhCH_2O)C_6H_3CO_2H$* (V), m. 122° (EtOH). V (5 g.) and 2.5 g. $H_2NCH_2CO_2H$ dissolved with warming in a soln. of 2.5 g. KOH and 2.5 g. K_2CO_3 in 40 cc. H_2O, the soln. refluxed 4 hrs. with a little Cu powder, filtered, dild. with 400 cc. H_2O, and acidified with concd. HCl yielded 5.7 g. *2,4-$HO_2C(PhCH_2O)C_6H_3NHCH_2CO_2H$* (VI), m. 221 (decompn.) (EtOH with H_2O). VI (15 g.) and 27 g. $Na_2CO_3.10H_2O$ in 300 cc. H_2O treated dropwise with shaking with 5.7 g. Ac_2O, shaken 1 hr., and acidified with concd. HCl to Congo red yielded 15.5 g. *N-Ac deriv.* (VII) of VI, m. 110° with previous sintering. VII (5 g.) treated in the usual manner with 25 cc. Ac_2O and 7 g. NaOAc yielded 4 g. *1,3-diacetyl-5-(benzyloxy)indoxyl* (VIII), m. 110° (EtOH). VIII (6 g.) added to 9.0 g. $Na_2SO_3.7H_2O$ in 180 cc. H_2O and 180 cc. EtOH at reflux temp., refluxed 1 hr., and allowed to stand overnight yielded 3.5 g. *1-acetyl-5-(benzyloxy)indoxyl* (IX), m. 170° (EtOH). $NCCH_2CO_2H$ (4.5 g.), 13.5 cc. xylene, and 1.5 g. IX added to 22.5 g. PhOH and 1.2 g. NH_4OAc and

(84 g.) and 8 g. NaOH in 100 cc. H$_2$O and 800 cc. EtOH refluxed 8 hrs. with 70 cc. PhCH$_2$Cl, treated dropwise with

Neutral oxalate (0.2 g.) of XI in 10 cc. abs. EtOH heated

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6
2-may

Costin D. Nenitzescu and Dan Răileanu

with 0.1 g. $(CO_2H)_2$ in EtOH, hydrogenated over 0.1 g. 10% Pd-C, and filtered, and the filtrate dild. with dry Et$_2$O pptd. 0.13 g. *acid oxalate* of *serotonin*, m. 198° (decompn.) (abs. EtOH-Et$_2$O). X (1 g.) in 50 cc. Me$_2$CO treated with 40 cc. 2% aq. NaOH and 10 cc. 30% H$_2$O$_2$, kept 24 hrs. at room temp., refluxed 5 min., evapd. *in vacuo*, and the residue cooled deposited 0.58 g. *5-benzyloxy-3-indolylacetamide*, m. 148°.

F. W. Hoffmann

CG
2/2

NENITZESCU, C. D.

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Distr: 4E3d

27

Mechanism of the hydride transfer in the Scholl reaction. Costin D. Nenitzescu and Alexander Balaban (Tech. Hochschule, Bucharest, Romania). *Chem. Ber.* 91, 2109-16 (1958).—Slight heating of 1-$C_{10}H_7OEt$ (I) with anhyd. $PhSO_3H$ (II) in $PhNO_2$ yields *(4-EtOC₁₀H₆)₂* (III) and *p-H₂NC₆H₄O₃SPh* (IV). IV was also obtained from PhNHOH and II in nonpolar solvents, indicating that PhNHOH is the primary reduction product from $PhNO_2$ in the reaction of I with II in $PhNO_2$. The hydride transfer from the dihydroaromatic intermediate formed in the Scholl reaction to the $PhNO_2$ (acting as acceptor) is catalyzed by II. The catalytic effect of II on the hydride transfer was demonstrated on the model system 9,10-dihydroanthracene (V) which with II in $PhNO_2$ yielded anthracene and IV; no hydride transfer occurred in the absence of II. The mechanism of the Scholl reaction is discussed. I (5 g.) in 52 g. $PhNO_2$ and 9.8 g. II heated 14 hrs. at 75°, cooled, and filtered gave 1.2 g. III, m. 113°; the filtrate steam-distd., and the resinous residue (about 3 g.) crystd. from C_6H_6-EtOH yielded an addnl. 1.0 g. III; the aq. steam distn. residue basified with NaOH gave 0.9 g. IV, needles, m. 101°; *N-Ac deriv.* of IV, m. 121° (aq. EtOH or C_6H_6-petr. ether); *N-PhSO₂ deriv.* of IV, m. 128° (C_6H_6-ligroine or aq. EtOH); coupling product with 2-$C_{10}H_7OH$, red needles, m. 157° (EtOH); *picrate* of IV, yellow plates, m. 173-4° (MeOH). 2-$C_{10}H_7OEt$ heated 20 hrs. at 80°, $(1-C_{10}H_7)_2$ heated 4 hrs. at 190°, and 1-$C_{10}H_7Bz$ heated 2 hrs. at 180° with II in $PhNO_2$ gave in all cases IV from the aq. phase, but only resinous products from the $PhNO_2$ soln. II (14 g.) in cold dry Et_2O treated dropwise with 10 g. PhNHOH in Et_2O deposited *PhNHO₃SPh*, needles, decomp. about 70°; a sample heated in a test tube on the H_2O bath underwent violent decompn. and formed tarry products. II (20 g.) in 1 l. C_6H_6 heated to boiling, treated dropwise with 7 g. PhNHOH in 200 cc. C_6H_6, heated 10 min., and cooled deposited 19 g. II salt of IV, m. 205-6°, which with alkali gave IV, m. 101°; picrate, m. 173°. A similar run with the reverse addn. of the reactants gave the $PhNH_2$ II salt and from the soln. PhN:N(O)Ph. V (5 g.) and 11 g. II in 50 cc. dry $PhNO_2$ heated 44 hrs. at 100° and cooled deposited 3 g. anthracene (1.1 g. 2nd crop, obtained by steam distn.); the aq. distn. residue treated with NaOH gave 1.3 g. IV, m. 101°. A similar run proceeded in the same manner during 1 month at 20°. V in $PhNO_2$ heated for a longer period of time at 100° without II gave only unchanged V; a similar run at 200° during 15 hrs. gave anthracene and small amts. of $PhNH_2$ and H_2O. V in C_6H_6 refluxed with II gave only unreacted V.

W. Hoffmann

LB

1/1

NENINTSESCU, K.

5(0) SOV/64-59-4-1/27

AUTHOR: Zhavoronkov, N. M., Deputy Chairman of the Organization Committee of the VIIIth Mendeleyev Congress, Corresponding Member of the AS USSR

TITLE: VIII. Mendeleyev Congress of General and Applied Chemistry (VIII. Mendeleyevskiy s"yezd po obshchey i prikladnoy khimii)

PERIODICAL: Khimicheskaya promyshlennost', 1959, Nr 4, pp 1-10 (USSR)

ABSTRACT: From March 16 to 23, 1959, the eighth traditional congress of the Russian chemists took place in Moscow. The congress was organized by the Academy of Sciences of the USSR, by the Vsesoyuznoye khimicheskoye obshchestvo imeni D. I. Mendeleyeva (All-Union Chemical Society imeni D. I. Mendeleyev), the Gosudarstvennyy komitet Soveta Ministrov SSSR po khimii (State Committee of the Council of Ministers of the USSR of Chemistry) and by the Ministerstvo vysshego obrazovaniya SSSR (Ministry of Higher Education of the USSR).

[redacted] In the first plenary meeting a contribution of the Chairman of the State Committee of the Council of Ministers of the USSR of Chemistry V. S.

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Fedorov was given about the "Problems of Scientific Technical Progress of the Chemical Industry" and by the Academician V. A. Kargin on the "Fundamental Problems of the Polymer Chemistry". In the plenary meetings the following contributions were given: Academician A. N. Nesmeyanov "The Periodic System of the Elements of D. I. Mendeleyev and Organic Chemistry", Academician N. N. Semenov - "Fundamental Problems of Chemical Kinetics", Academician V. I. Spitsyn -"Modern State of the Periodic System of D. I. Mendeleyev", Academician A. P. Vinogradov - "Fundamental Problems of Radiochemistry", Academician V. A. Engel'gardt - "Fundamental Problems of Biochemistry", Professor A. V. Sokolov - "Chemical Problems of the Agriculture of the USSR", Director of the Nauchno-issledovatel'skiy institut khimicheskogo mashinostroyeniya (Scientific Research Institute of Chemical Machine Construction) V. V. Nikolayev - "Main Problems of the Chemical Apparatus and Machine Construction", Corresponding Member of the AS USSR Ya. K. Syrkin - "Present State of the Problem of Valence" and Academician A. P. Aleksandrov - "Chemical Aspects in the Application of Atomic Energy".

[redacted] Liu Ta-kang, Director of the Institute of Chemistry of the Academy of Sciences of the Chinese People's Republic - "State of the Studies on Rare Elements in the Chinese People's Republic", K. Nenitescu, Rumanian scientist, Academician - "Separation of the Complex of Cyclobutadione With Silver Nitrate", Academician I. Murgulescu -"Kinetics of the Dehydration of the Crystal Hydrates", and Academician R. Ripan - "Investigation of the Structure of Some Inorganic Compounds by Means of Radioactive Isotopes", Academician Geza Shay, President of the Hungarian Chemical Society - "Quantitative Ratio in Frontal Gas Chromatography". K. K. Ingold, Professor of the London University - "Nitration With Respect to Nitrogen and Oxygen", R. P. Bell, President of the Faraday Society and Professor at Oxford University - "The

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Tunnel Effect in Reactions With the Action of Hydrogen Isotopes" Ch. K. Ingol'd made a lecture in Leningrad on the theme "Studies of the Kinetics of the Nitration Process",

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NENITZESCU, C.D.

4E3d

2gg(HB)

Isomerization of 1,2-*trans*-dibromocyclohexane under the influence of anhydrous aluminum bromide. Costin D. Nenitzescu and Ilie G. Dinulescu. *Acad. rep. populare Romîne, Studii cercetări chim.* 7, 7-14(1959).—Halogen migration in dihalocyclohexanes was investigated following previous work (*C.A.* 37, 3740^4) on H$^+$ migration in Friedel-Crafts type reactions. Present work concerned behavior of dibromocyclohexanes in the presence of anhyd. $AlBr_3$ at 0° in a nonpolar inert medium, CS_2. 1,2-*trans*-Dibromocyclohexane isomerized to yield 70-87% isomeric product contg. approx. 25% cryst. *trans*-1,4-$C_6H_{10}Br_2$ and 75% liquid, mostly 1,3-$C_6H_{10}Br_2$ (probably trans), dissolving partly the 1,4-isomer. No residue of initial 1,2-isomer was detected. The same final equil. compn. was obtained with *trans*-1,2-$C_6H_{10}Br_2$, 1,3-$C_6H_{10}Br_2$ (probably trans), and *trans*-1,4-$C_6H_{10}Br_2$ as initial isomers. The reaction conformed to carbocation formation, under influence of the electrophilic catalyst, followed by H$^+$ migration. Similar work with a polar medium, HBr, gave different results (cf. Goering and Sims).

M. Lapidot

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NENITZESCU, C.D.

Distr: 4E3d/4E3b/4E2c(j)

Cyclobutane series. V. Some bromo derivatives of cyclobutane. Margareta Avram, Ilie Marica, and C. D. Nenitzescu (Acad. R.P.R., Bucharest, Romania). *Acad. rep. populare Romîne, Studii cercetări chim.* 7, 155-67(1959) (German summary); cf. *C.A.* 53, 16993*f*; preceding abstr.—Both *cis*- (I) and *trans*-1,2-cyclobutanedicarboxylic acid (II) were converted into a mixt. of *cis*- (III) and *trans*-1,2-dibromocyclobutane (IV) by the Hunsdiecker reaction (which was therefore not stereospecific). I (36 g.), m. 139.5-40.5°, or its anhydride (V) in 150-200 cc. H_2O was added to 85 g. $AgNO_3$ in 1700 cc. H_2O. The Ag salt of I (80-85 g.) pptd. The salt was dissolved in 1100 cc. dry CCl_4, stirred, and cooled to 0°. Br (87.9 g.) was added (the temp. rose to 65°), the soln. filtered, 3% aq. $NaHSO_3$ added till discoloration occurred, the soln. washed (H_2O), dried ($MgSO_4$), the solvent evapd. in a column, and the residue fractionated twice *in vacuo* to give a mixt. (19.2-21.6 g.) of 14% III and 78% IV. II, m. 131-1.5°, was prepd. from the di-Et ester of I, b_{15} 138°, by converting it with NaOEt to the di-Et ester of II, b_{15} 120°, and then boiling with 6*N* HCl 12 hrs. The Ag salt of II was converted into a mixt. of 26% III and 65% IV as above. 1-Bromocyclobutene was dissolved in 130 cc. isopentane and dry HBr was introduced at 0° under ultraviolet illumination 3-4 hrs. Fractionation gave 10 g. III, 97% purity, b_{45} 102°, m. −14.5 to −14.2°, d_{25} 2.0103, n_D^{25} 1.54981. IV, 97% purity, b_{45} 93-4°, m. −3.5 to −4.0°, d_{25} 1.93651, n_D^{25} 1.53486, was prepd. in $CHCl_3$ by bromination of cyclobutane. A mixt. (21.4 g.) of III and IV, 21.4 g. $AlBr_3$, and 0.036 cc. H_2O was stirred in 160 cc. CS_2 at 0-15° 10 hrs. The CS_2 soln. was decanted from the red resin which sepd., washed, evapd., and the residue fractionated *in vacuo*. Thus, 15-16 g. mixt. was obtained contg. 19% III, 47.5% IV, and 33.5% 1,3-dibromocyclobutane (VI) (detd. by gas chromatography). This mixt. (10.7 g.) and 20 g. powd. KOH was heated at 100-10° 1 hr. From the gas evolved, 1.4 cc. $HC:CCH:CH_2$ (VII) condensed in a trap, and $HC:CH$ was adsorbed in a Cu(I) soln. VII was identified by converting it into tetrabromovinylacetylene. A mixt. (10.7 g.) of III and IV dissolved in 10 cc. CS_2 was mixed with 3 g. $AlBr_3$ dissolved in 80 cc. CS_2 at 0°, satd. with dry HBr, agitated 2 hrs. at 0° and 6 hrs. at 15°. The solvent was decanted, evapd., and the residue distd. *in vacuo*. Tribromobutane (VIII) (10 g.), b_6 104-5°, d_{25} 2.2028, n_D^{25} 1.56368, was obtained. VIII (2 g.) heated at 110° with 5 g. KOH gave VII. 1,2-Dibromocyclobutane-1,2-dicarboxylic acid (IX) (30.3 g.) was dissolved in 200 cc. H_2O, neutralized with NH_3 to pH 7, and dropped into a soln. of $AgNO_3$ (34.5 g. in 800 cc. H_2O). The Ag salt of IX (47-50 g.) pptd. The Hunsdiecker reaction of this (18.8 g.) gave 3 g. 1,1,2,2-tetrabromocyclobutane (X), m. 126° (MeOH). $AlBr_3$ (1.36 g.) dissolved in 100 cc. CS_2 was added to 18.6 g. X at 0° and agitated at 10-20° 10 hrs. A mixt. of isomers ($C_4H_4Br_4$) was obtained, b_2 114-15°. I (7.2 g.) dissolved in 60 cc. C_6H_6 was mixed with 7.9 g. dry pyridine and 22.2 g. $Pb(OAc)_4$, and stirred at 50-60° 1 hr. Cyclobutene (XI) evolved was introduced into a soln. of Br in CH_2Cl_2, from which 1.6 g. 1,2-dibromocyclobutane was obtained, b_{80} 95-100°. A flask contg. 150 cc. dry CCl_4, 17 g. bromosuccinimide, and 0.5 g. benzoyl peroxide was filled with carefully dried XI and agitated at 70-4° 6 hrs. The CCl_4 soln. was decanted from the polymer (oil) sepd. and the solvent evapd. By fractionating the residue, 1,2-dibromocyclobutane (2.8-3.5 g.), b_{30} 80-5°, was obtained.

VI. The decomposition of quaternary salts of 1,3-diaminocyclobutane. Margareta Avram and C. D. Nenitzescu. *Ibid.* 169-74.—The attempted Hoffmann degradation of 1,3-bis(dimethylamino)cyclobutane dimethobromide (I) in the presence of PhLi (II) was found to be anomalous, giving phenylcyclobutene (III). I (13 g.), m. above 265°, AVRAM, MARGARETA; MATEESCU, GHE; and NENITZESCU, C.D.

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was prepd. from 9.5 g. 1,3-bis(dimethylamino)cyclobutane (IV) dissolved in 10 cc. dry MeOH at 0° by satg. it with dry MeBr and allowing the mixt. to stand 12 hrs. To 100 cc. Et_2O soln. of II, obtained from 9.6 g. PhBr, 4.9 g. I was added. The soln. was agitated 80-100 hrs. Me_4N was isolated as its picrate (3 g.). Also obtained were a white cryst. product, m. 157° (EtOH), and 0.45 g. III, b_6 80-8°. III was converted into 2,4-dinitrophenyl phenylcyclobutenyl sulfide, m. 98°. IV (3.6 g.) was added to 15 cc. 30% H_2O_2 and stirred at 0-10° 10 hrs. to give 1,3-bis(dimethylamino)cyclobutane *N,N'*-dioxide, m. 85-90° (hygroscopic); picrate m. 186° (MeOH). T. ~~[illegible]~~

NENITZESCU, C. D.

Distr: 4E3d

1

Addition compound of phenylcycloöctatetraene with dimethyl acetylenedicarboxylate and with benzoquinone. I. Pogány, Gh. Mihai, and C. D. Nenitzescu (Acad. R.P.R., Bucharest, Romania). *Acad. rep. populare Romîne, Studii cercetări chim.* 7, 235-42(1959)(German summary).—Phenylcycloöctatetraene (I) (5 g.) was heated with 6 g. di-Me acetylenedicarboxylate (II) at 130° 48 hrs. under a CH_4 atm. Fractionation *in vacuo* gave an addn. product (III), yellowish sirup, and a $CHCl_3$-sol. black resinous residue. III (5 g.) was kept under a CO_2 atmosphere at 280°. Gas (60 cc.) was evolved; the residue was fractionated *in vacuo*. Di-Me phthalate (1.35 g.), b_2 110-12°, and a nonvolatile polymer of phenylcyclobutadiene were obtained. The latter was converted into benzoic acid by oxidn. with $KMnO_4$. I (8.5 g.) and benzoquinone (9 g.) was boiled in 125 cc. C_6H_6 under an inert atm. 24 hrs. From the solid residue obtained by removing C_6H_6 *in vacuo*, 11 g. IV, m. 174-76°, was extd. with EtOH and recrystd. m. 184-6° (EtOH). IV (11 g.) was boiled in 100 cc. Ac_2O and 5 cc. concd. HCl 3 hrs., poured into 400 cc. H_2O, and agitated 1 hr. The diacetoxy deriv. (V) of IV (12.5 g.), not described so far, was obtained, m. 154-6° (recrystd. from EtOH, white needles, m. 157°). V (2 g.) was heated to 280-90°. Sublimation *in vacuo* at 130-80° gave 0.85 g. 1,4-diacetoxynaphthalene, m. 126-7° (MeOH), and a nonvolatile polymer, which was converted into benzoic acid as above. It was concluded that the structures of III and IV were as indicated. The Ph substituent in both III and IV was either at the 2- or at the 8-position. The thermal decompn. of III and IV followed the Alder-Rickert scheme.

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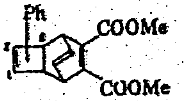
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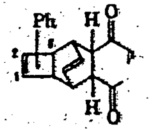
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T. Szélli

Distr: 4E2c(j)/4E3b/4E3d

1

Preparation of cyclobutadiene. C. D. Nenitzescu, Margareta Avram, Eliza Marcia, Maria Maxim, and Doina Dinu (Chem. research center, Acad. R.P.R., Bucharest, Romania). *Acad. rep. populare Romîne, Studii cercetări chim.* 7, 481-504(1959).—A literature review (and results of exptl. work based on it) was presented in connection with the synthesis of cyclobutadiene or alkyl- and aryl-cyclobutadiene derivs. Hofmann degradation of the diquaternary base of 1,3-diaminocyclobutane, thermal decompn. of cyclooctatetraene adducts, and attempts to prepare cyclobutadiene from brominated derivs. of cyclobutane were performed. Cyclobutadiene was detected in $AgNO_3$ and Hg complexes, as well as (for a short period) in the gaseous state. Transformation of the Hg-cyclobutadiene complex into cyclooctatetraene was accomplished. Alkylated and arylated derivs. of cyclobutadiene were prepd. (32 references) (Summaries in Russian and English).

M. Lapidot

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1-BW(BW)

2-Jog(NB)[illegible]

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Distr: 4E2c(j)/4E3d

A new rearrangement of phenyl alkanes. Rearrangements of diphenyl butanes. Costin D. Nenitzescu and Alice Glatz (Chem. research center, Acad. R.P.R., Bucharest, Romania). *Acad. rep. populare Romîne, Studii cercetari chim.* 7, 505-19(1959).—The 1,1-, 1,2-, 1,3-, and 2,2-diphenylbutanes, the 1,1-, and 2,3-dichlorobutanes, the 1,2-, 1,4-, and 2,3-dibromobutanes, and the phenylbromobutanes (1-phenyl-1-bromo, 1-phenyl-2-bromo, 1-bromo-2-phenyl, 1-phenyl-3-bromo, 1-bromo-3-phenyl, 1-phenyl-4-bromo, and 2-phenyl-2-bromo) were prepd. and refluxed with $AlCl_3$ in C_6H_6 soln. Intramol. rearrangements took place, both in the alkylating agent before the Friedel-Crafts reaction, and in the diphenylbutanes produced in the reaction. In the latter rearrangement, phenyl groups tended to migrate toward or persist in the 1-position, in agreement with previous observations made in the monophenylalkane series. Therefore, in most of the latter rearrangements, the product was mostly 1,1-diphenylbutane; second in order of stability was *meso*-2,3-diphenylbutane. (28 references). (Summaries in Russian & English).

M. Lapidot

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1-BW (BW)

1-MJ (NB)

1-Jag (MAJ)

2

1/1

Distr: 4E2c(j)/4E3d

The decarbonylation of 1,4-dihydro-1-naphthoylchloride
and its implications in the mechanism of the Scholl and
Gattermann-Koch reactions. Alexandru T. Balaban and
C. D. Nenitzescu (Org. chem. lab., Polytech. inst.,
Bucharest, Romania). *Acad. rep. populare Romîne, Studii*
cercetări chim. 7, 521-9(1959).—1,4-Dihydro-1-naphthoic
acid (prepd. from 1-naphthoic acid and 1% Na-Hg) and
its derivs., the chloride (the acid with excess $SOCl_2$), the
amide (C_6H_6 soln. of the chloride and NH_3 gas), the anilide
(the chloride and aniline in abs. ether), and the nitrile
(the amide and PCl_5), were prepd. Treatment of the
chloride with $AlCl_3$ in CS_2 caused its decompn. into HCl,
CO, and naphthalene, with formation of 2,2'-binaphthyl;
the latter was formed in larger amts. (together with higher
mole. wt. polymers) if naphthalene was present from the start.
If the CS_2 was replaced by anisole or toluene, Friedel-
Crafts reactions occurred with these solvents to yield a
mixt. of *o*- and *p*-anisyl 1,4-dihydro-1-naphthyl ketone and
a mixt. of *o*- and *p*-tolyl 1,4-dihydro-1-naphthyl ketone.
This confirmed the mechanism previously proposed for the
Scholl reaction and had a bearing on the mechanism of the
Gatterman-Koch reaction. (32 references) (Summaries in
Russian and English). M. Lapidot

4
1- BW(BW)
2- JoJ(NB)(may)
2-

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2l

IOAN, V.; TEODORESKU, L. [Teodorescu, L.]; TSITSEYKA, Sh. [Titeica, S.], acad.; NENITSESKU, K.D. [Ninitescu, C.D.], acad.

Mechanism of the reaction of alkylation of the aromatic ring. III. Catalytic action of benzenesulfonic acid in the reaction of benzyl chloride with benzene. IV. Alkylation with various benzyl halides. Rev chimie 4 no.2:171-187 '59. (EEAI 9:7)

1. TSentr Khimicheskikh issledovaniy Akademii RNR, Otdel Organicheskoy khimii, Bukharest. 2. Redacteur en chef, Comite de redaction, Revue de Chiimie (for Nenitescu)
(Benzene) (Alkylation) (Aromatic compounds)
(Ring compounds) (Catalysts) (Benzenesulfinic acid)
(Benzyl group) (Halides) (Chlorotoluene)

AVRAM, Margareta; MARICA, Elisa; NENITZESCU, Costin D.[Nenitescu, C.D.], acad.

Research in the series of cyclobutane. V. On the bromoderivatives of cyclobutane. VI. Research in the obtainment of quaternary salts derived from cyclobutanediamine. Rev chimie 4 no.2:253-270 '59.
(EEAI 9:7)

1. Aus dem Chemischen Forschungslaboratorium der Akademie der RVR, Bukarest. 2. Redecteur en chef, Comite de redaction, Revue de Chimie; Mitglied der Akademie der Rumanischen Volksrepublik (for Nenitescu)
(Ring compounds) (Cyclobutane) (Bromine)
(Quaternary compounds) (Salts) (Cyclobutanediamine)

NENITESCU, Costin D., acad.; AVRAM, Margareta; MARICA, Eliza; MAXIM, Maria;
DINU, Doina

Tests for obtaining the cyclobutadiene. Studii cerc chim 7 no.4:
481-504 '59. (EEAI 9:7)

1. Comitetul de redactie, Redactor responsabil, Studii si cercetari de chimie (for Nenitescu). 2. Centrul de cercetari chimice al Academiei R.P.R., Sectia de chimie organica, Bucuresti.
(Mercury) (Cyclobutadiene) (Alkyl groups)
(Aryl groups) (Silver nitrate) (Complex compounds)

NENITESCU, Costin D.; GLATZ, Alice.

A new regrouping of phenylalkanes: regrouping in the diphenylbutane series. Studii cerc chim 7 no.4:505-519 '59. (EEAI 9:7)

1. Centrul de cercetari chimice al Academiei R.P.R., Sectia de chimie organica, Bucuresti.
(Phenyl group) (Paraffins) (Diphenylbutane)
(Aluminum chloride)

BALABAN, Alexandru T.; NENITESCU, Costin D., acad.

On the decarboxylation of the 1,4-dihydro-1-naphthalenecarboxylic acid, and its effects on the mechanism of the Sscholl and Gattermann-Koch reactions. Studii cerc chim 7 no.4:521-529 '59. (EEAI 9:7)

1. Institutul politehnic, Bucuresti, Laboratorul de chimie organica.
(Dihydronaphthoic acid) (Carboxyl group)
(Aluminum chloride)

G-2

COUNTRY : Rumania

CATEGORY : Organic Chemistry--Organic synthesis.

ABS. JOUR. : RZKhim., No. 5 1960, No. 17743

AUTHOR : Nenitescu, C. D. and Badea, F.

INST. : Rumanian Academy of Sciences

TITLE : On the Catalytic Dehydrogenation of Cyclooctatetraene

ORIG. PUB. : Comun Acad RPR, 9, No 3, 245-248 (1959)

ABSTRACT : Experiments with the dehydrogenation of cyclooctatetraene by passing it through heated Pd/C gave unidentified gaseous products and ethylbenzene in the place of the expected dimers of benzocyclobutadiene.

V. Skorodumov

CARD: 1/1

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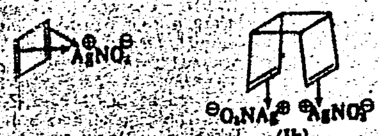
NENITZESCU C. D.

Studies in the cyclobutane series. IV. A silver nitrate complex $C_4H_4.AgNO_3$. Margarete Avram, Elise Marica, and Costin D. Nenitzescu (Acad. R.P.R., Bucarest, Romania). ~~Chem. Ber. 92,~~ 1088-91(1959); cf. *C.A.* 52, 18316g.—The elimination of Br from 1,2,3,4-tetrabromocyclobutane (I) with Li-Hg yielded an unstable Hg deriv. which with $AgNO_3$ gave a cryst. complex having the structure Ia or Ib. Cyclooctatetraene dibromide oxidized in Me_2CO with $KMnO_4$ at −5° by the method of Cope and Burg (*C.A.* 47, 6925f) gave 43% *3,4-dibromocyclobutane-cis-1,2-dicarboxylic acid* (II), m. 191-2° (Et_2O-petr. ether). II



(Ia) (Ib)

(41.2 g.) in 100 cc. H_2O adjusted with 3% aq. KOH to pH 7, treated dropwise with stirring with 46.5 $AgNO_3$ in 900 cc. H_2O, and the ppt. filtered off rapidly, washed with H_2O, EtOH, and Et_2O, and dried 24 hrs. in the dark over P_2O_5 gave 64 g. *Ag salt* (III) of II. III (40 g.) and 600 cc. CCl_4 refluxed with the slow removal of 30-40 cc. CCl_4 through the condenser, the residue cooled, treated with 8.1 cc. Br, heated briefly on the water bath, refluxed 15 min., cooled, filtered, washed, dried, and evapd., and the residue (9.15 g.) dissolved in dry Et_2O, kept 24 hrs. over K_2CO_3, and evapd. gave I, b_3 115-20°, m. 104° (MeOH or petr. ether). I (1.87 g.) and 42 g. 0.5% Li-Hg shaken 15-20 hrs. at room temp. with 35 cc. abs. Et_2O, the Et_2O layer decanted, the Hg sludge washed with Et_2O, the combined exts. washed, dried, and evapd. under N gave a pale yellow oil with an acrid, characteristic odor, decomp. about 60°. The Et_2O soln. from a similar run shaken 1-2 min. with satd. aq. $AgNO_3$ and filtered, and the residue (0.52 g.) washed with Et_2O and recrystd. from EtOH gave Ia-Ib, m. 140°. Ia-Ib (0.22 g.) treated under CO_2 with dil. NH_4OH gave 0.3 cc. gas. Ia-Ib (0.52 g.) powdered and added gradually to Br in CH_2Cl_2, and the mixt. filtered, washed, dried, and evapd. gave 0.1 g. oil, contg. 42.3% Br and 7.15% N, which decompd. on attempted distn. *in vacuo*. The formation of cyclobutadiene which existed for short periods of time in the gaseous state was demonstrated by treating Ia-Ib with a small amt. of steam and sweeping with air through Ascarite and $Mg(ClO_4)_2$ to give a gas which formed Ia-Ib with $AgNO_3$ soln.

F. W. Hoffmann

5
2-May
4E2c

BALABAN, A.T.; NENITSESKU, K.D.

Ultraviolet absorption spectrum of pyrylium chlorate. Izv. AN SSSR.Otd. khim. nauk no.11:2064-2065 N '60. (MIRA 13:11)

1. Politekhnicheskiy institut i Institut atomnoy fiziki, Bukharest.
(Pyrylium compounds—Spectra)

CIORANESCU, Ecaterina; BUCUR, Aurora; BANCIU, M.; NENITESCU, C.D.

Carbonium ion reactions. Pt. 1. Studii cerc chim 14 no.2:127-134 F '65.

1. Institute of Organic Chemistry, Rumanian Academy, Bucharest. Submitted November 17, 1964.

NENITZESCU, C. D.

Distr: 4E2c(j)/4E3d

6
7-Bul(BW)
2-JAJ(NB)(MAY)
2

Dichlorocarbene in the pyrolysis of silver trichloroacetate. Viorica Ioan, F. Badea, Ecaterina Cioranescu, and C. D. Nenitzescu (Akad. Bucharest, Romania). *Angew. Chem.* 72, 416(1960); cf. preceding abstr.—The following mechanism, postulating dichlorocarbene as intermediate, was suggested to account for the formation of $(Cl_3CCO)_2O$ in the pyrolysis of Cl_3CCO_2Ag (I): I → $:CCl_2 + CO_2 + AgCl$; $:CCl_2$ + I → $(Cl_3CCO_2CCl_2)^-Ag^+ \rightarrow Cl_3CCOCl + CO + AgCl$; Cl_3CCOCl + I → $(Cl_3CCO)_2O + AgCl$. Gas analysis confirmed the formation of equimolar amts. of CO_2 and CO. I heated in cyclohexene at 70° yielded the anhydride as major product, about 10% dichloronorcarane (identified by gas chromatography), and a considerable amt. of 1-formylcyclopentene. S. Altergut

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CHIORENESKU, Ekaterina [Cioranescu, Ecaterina]; BUKUR, Avrora [Bucur, Aurora];
MAKSIM, Mariya [Maxim, Maria]; [redacted] [Nenitescu, C.D.],acad.

A new method of separating thiourea from ammonium thiocyanate. Rev chimie 5 no.2:223-226 '60. (EEAI 10:4)

1. TSentr khimicheskikh issledovaniy Akademii RNR, Otdel organicheskoy khimii, Bukharest. 2. Akademiya nauk RNR, chlen Akademii nauk RNR, Comite de redaction, Revue de chimie, membre de l'Academie de la Republique Populaire Roumaine, redacteur de chef (for Nenitescu)
(Thiourea) (Ammonium thiocyanate)

CIORANESCU, Ecaterina; BUCUR, Aurora; MAXIM, Maria; NENITESCU, C.D., acad.

A new method in separating thiourea from ammonium thicyanide. Studii cerc chim 8 no.1:67-71 '60. (EEAI 9:8)

1. Centrul de cercetari chimice al Academiei R.P.R., Sectia chimie organica, Bucuresti. 2. Comitetul de redactie, Studii si cercetari de chimie (for Cioranescu). 3. Redactor responsabil, Studii si cercetari de chimie (for Nenitescu).

(Thiourea) (Ammonium thiocyanate)
(Carbon tetrachloride)

BALABAN, Aleksandru T.; GENYA, Anisiya [Genea, A.]; NENITSESKU, Kostin, D. [Nenitzescu, C.D.]

Preparation of pyrylium salts by bis-acylation of olefins. Report 5: Bis-acylation of di- and triisobutylene. Izv.AN SSSR,Otd.khim.nauk no.6:1102-1107 Je '61. (MIRA 14:6)

1. Politekhnicheskiy institut, Bukharest.
(Propene) (Acylation)

NECSOIU, Ileana; NENITZESCU, C. D. [Nenitescu, C.D.]

Chromic acid oxidation. Mechanism of oxidation of 1,2-diphenylethane and 1,2-diphenypropane. Rev chimie 6 no.2:245-257 '61.

1. Chemical Institute of the Academy of the R.P.R., Bucharest, Organic Chemistry Section. 2. Membre du Comité de rédaction et rédacteur en chef, "Revue de chimie, "membre de l'Academie de la Republique Poupulaire Roumaine (for Nenitzescu)

NEKSHOYU, Ilyana [Necsoiu, Ileana]; NENITSESKU, K.D. [Nenitescu, C.D.]

Rearrangement of phenylindans under the action of aluminum chloride. Rev chimie 6 no.2:259-268 '61.

1. Nauchno-issledovatel'skiy institut khimii Akademii nauk RNR, Bukharest, otdel organicheskoy khimii 2. Membre du Comité de rédaction et rédacteur en chef, "Revue de chimie" (for Nenitescu)