"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

PAKHOMOV, I. I., TERENTIYEV, A. G.

Medical Instruments and Apparatus - Periodicals

About the Journal "Meditsinskaia promyshlennost8 SSSR" (Pharmaceutical and medical supplies industry of the U.S.S.R."). Med. prom. No. 3, 1952.

9. Monthly List of Russian Accessions, Library of Congress, August, 1952. 1953, Uncl.

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TERENT'YEV, A. G.: GRIGV, G. T.

Medical Sup lies

Application of the method of statistical control of quality of products manufactured at plants producing medical equipment and supplies. Med. prom., No. 4, 1952.

9. Monthly List of Russian Accessions, Library of Congress, November, 1952 1953, Uncl.

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

ACCESSION NR: AR3002670 S/0124/63/000/005/B042/B042

SOURCE: Rzh. Mekhanika, Abs. 5B216

AUTHOR: Terent'yev, A.G.

TITLE: On the theory of a circular wing

CITED SOURCE: Sb. aspirantsk. rabot, Kazansk. un-t. Tochnye n. Kazan', 1962, 203-215

TOPIC TAGS: wing, flow, irrotational flow, circular wing, vortex layer, source, sink, trigonometric series, substitution, zhukovskiy profile

TRANSLATION: The steady state irrotational flow of an incompressible ideal liquid around a circular wing at a small angle of attack is considered. The average wing cross section differs little from the rectangular. The wing is replaced by a system of connected vortex rings situated along a cylindrical surface. On the basis of the work of S.M. Belotserkovskiy (Tr. VVIA, 1953, no. 452) and Weissinger (Weissinger J., Z. Flugwiss., 1956, 4, no. 3-4, 141-150 Rzh Mekh, 1958 no. 1, 329) the integral relations connecting the components of the induced

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L 15731-63
ACCESSION NR: AR3002670

velocity on the cylindrical surface with the intensity of the vortex layer w found. Using the usual substitution of variables for the transition to trigo nometric series. For the calculation of the effect of the thickness of the s tion the wing is replaced by the system of sources and sinks, situated on the cyl ri- cal surface, and the problem is solved by a similar method. Graphs of the auxiliary functions are provided. Two examples are considered -- a thin cylindri- cal wing and a wing with a symmetric Zhukovskiy profile. Bibl. 4 names. A.I. Borisenko

DATE ACQ: 14Jun63 SUB CODE: AI ENCL: 00

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ACCESSION NR: AR4034728 S/0124/64/000/003/B052/B052

SOURCE: Ref. zh. Mekhan., Abs. 3B320

AUTHOR: Terent'yev, A. G.

TITLE: Problem of jet flowby of two plates under a free surface

CITED SOURCE: Sb. Itog. Nauchn. konferentsiya Kazansk. un-ta za 1962 g. Sekts. matem. n.. Kazan', Kazansk. un-t, 1963, 179-181

TOPIC TAGS: hydrodynamics, hydraulics, flowby, jet stream flowby, mathematical solution

TRANSLATION: A plane problem is presented on flowby with interruption by a jet of weightless ideal noncompressible liquid past two plates. A jet of liquid of finite thickness is thrust past the first plate, which is set at an arbitrary angle to the flow. The second plate covers its part of the lower free surface. The depth of the flow is infinite.

The problem is solved by the reflection of the areas of change of the complex potential and complex speed on a rectangle. Expressions for the forces acting on

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the plates have been found. The work has an errata table.

DATE ACQ: 02Apr64 SUB CODE: AI ENCL: 00

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

TERENT'YEV, A.G. (Kazan')

Flow past a slanted plate in a channel according to a scheme with parallel walls. Izv. vys. ucheb. zav.; mat. no.3:155-168 '65. (MIRA 18:7)

APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6"

"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

L 45398-65 EWT(1)/EWP(m)

ACC NR: AR6016615 SOURCE CODE: UR/0044/65/000/012/B069/B069

47B

AUTHOR: Terent'yev, A. G.

TITLE: Problem of the braid of flow about a curvilinear arc with developed cavitations

SOURCE: Ref. zh. Matematika, Abs. 12B370

REF SOURCE: Tr. Seminara po obratn. krayev, zadacham. Kazansk. un-t, vyp. 2, 1964, 187-200

TOPIC TAGS: incompressible fluid, cavitation, small parameter

ABSTRACT: The author considers steady state plane cavitational flow about a curvilinear arc of given form by an ideal incompressible and weightless fluid. The problem is reduced to the solution of an integro-differential equation, generalizing the known theoretical jet equation of Villya [Will?]. The solution of this equation is constructed in the form of a series in powers of a small parameter which is proportional to the curvature of the arc and the cavitation number. Convergence of this series is proved for sufficiently small values of the parameter of decomposition. In the construction of the solution and in its investigation a method was used which is analogous to that used by the reviewer in the case of the Villya [Will?] equation (Dokl. AN SSSR, 1935, 3 (VIII), No. 4 (64)). Ya. Sekerzh-Zen'kovich [Translation of abstract]

UDC: 517.9:533.011

Card 1/1 hs SUB CODE: 20

APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6"

"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

L 25635-66 EWT(1)/EWP(m)/EWP(w)/EWA(d)/EWA(1) EM

ACC NR: AP6016103 SOURCE CODE: UR/0140/65/000/003/0155/0168

AUTHOR: Terent'yev, A. G. (Kazan')

ORG: none

TITLE: Flow around an inclined plate in a channel with parallel walls

SOURCE: IVUZ. Matematika, no. 3, 1965, 155-168

TOPIC TAGS: cavitation, flow analysis, planar flow

ABSTRACT: In this article the planar problem of flow around an inclined plate in a channel with parallel walls according to the Ahukovskiy-Roshko diagram is investigated. Formulae are presented for calculation of the buoyant force and resistance of the plate, along with formulae for determination of the elements of geometric flow. The following special cases of the problem are considered: cavitation flow around the disc close to the wall and in a limited flow, flow around the disc, according to N. Kirchhoff, in a channel and close to the wall. The application of the aspects of the problem investigated to the study of hydrofoils is mentioned. Orig. art. has: 5 figures and 8 formulas. [JPRS]

SUB CODE: 20 / SUBM DATE: 14Dec64 / ORIG REF: 004 / OTH REF: 001

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

L 04208-67 EWT(d)/EWT(1)/EWP(m) IJP(c) WW

ACC NR: AR6000710 SOURCE CODE: UR/0124/65/000/009/B056/B056

58

B

AUTHOR: Terent'yev, A. G.

TITLE: On the theory of flow around of a curvilinear arc by a scheme with a "mirror"

SOURCE: Ref. zh. Mekhanika, Abs. 9B362

REF SOURCE: Sb. Itog. Nauchn. konferentsiya Kazansk. un-ta za 1963 g. Sektsmatem., kibernet. i teoriya veroyatn., mekhan. Kazan', 1964, 120-122

TOPIC TAGS: streamline flow, function theory, complex function, cavitation, parametric equation, integrodifferential equation, convergent series

ABSTRACT: The two-dimensional problem of symmetric flow around a curvilinear arc of a given form by a stream of ideal incompressible and weightless fluid according to a scheme with a "mirror" (Ryabushinskiy scheme) is solved by the method of functions of a complex variable. According to this scheme, the jets that separate from the flown-around arc and that make up the boundary of the cavity, convert to a curvilinear arc, which closes the cavity and is a mirror image of the given arc. The solution is done in parametric form by the introduction of the parametric plane of the complex variable $u = \xi + i\tau$, on which the flow region corresponds to the interior of a rectangle with sides π and $-\pi i\tau$. An integro-differential equation is derived for the function $\theta(\xi)$, which is equal to the slope of the tangent to the contour with the x axis. This equation generalizes the known (in jet theory) Will equation, and it is solved under

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the assumption that the curvature of the airfoil profile is an analytic function of θ at $\theta = \pi/2$. The solution is constructed in the form of a series in powers of the small parameter λ, which is proportional to the curvature. It is indicated that the convergence of the series and the uniqueness of the solution of the problem were studied. A method of determining the geometric elements, the velocity, and the resultant force acting on the contour is indicated. It is observed that, by assigning two parameters, it is possible to calculate a unique arc which is some part of the curve in question, to calculate the cavitation number σ, and the drag coefficient of the arc taken over its midsection. Asymptotic formulas are given for the flow elements at small values of the determining parameters. Flow around an arc of a circle is examined in conclusion. Ya. I. Sekerzh-Zen'kovich [Translation of abstract]

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

L 00791-67 EWP(m)/EWP(k)/EWT(1)/EWT(m)/T-2/EWP(w)/EWP(v) IJP(c) EM

ACC NR: AR6014922 SOURCE CODE: UR/0124/65/000/011/B067/B067

70
B

AUTHOR: Terent'yev, A. G.

TITLE: The problem of oblique flow around a curvilinear arc with developed cavitation

SOURCE: Ref. zh. Mekhanika, Abs. 11B457

REF SOURCE: Tr. Seminara po obratn. krayev. zadacham. Kazansk. un-t, vyp. 2, 1964, 187-200

TOPIC TAGS: streamline flow, cavitation, convergent series, approximate solution, ideal fluid, incompressible fluid, parameter, conformal mapping

ABSTRACT: Steady-state plane cavitation flow of a stream of an ideal incompressible and weightless fluid around a cuvilinear arc of a given form according to a scheme with central symmetry is examined. According to this scheme, the domain bounded by the arc streamed around by jets, which are detached from the ends of the arc and which come together at a finite distance from it, is a domain with central symmetry. The problem is solved by the method of the theory of functions of a complex variable with the use of conformal mapping and an auxiliary parameteric complex plane. The problem reduces to the solution of an integro-differential equation, which generalizes the Will equation in the theory of jets. The solution of this equation, which is

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equal to the angle formed by the axis of the abscissas with the tangent to the arc, is constructed in the form of a series in powers of a small parameter, which is proportional to the curvature of the arc and the cavitation number. The convergence of this series is demonstrated for sufficiently small values of the parameter of the expansion. A method similar to that used in the case of the Will equation (Dokl. AN SSSR, 1935, 3 (8), No. 4 (64)) was used in constructing and studying the solution. Approximate formulas are given for determining the flow according to the arc parameters and for calculating the lift and drag. These formulas are given for arcs with small curvature and for small cavitation numbers (so-called developed cavitation). The cavitation flow around an arc of a circle is studied in more detail. Bibliography of 4 citations. Ya. I. Sekerzh-Zen'kovich [Translation of abstract]

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

TERENT'YEV, Aleksey Ivanovich; TRUBIN, M.I., red.; POD"YEL'SKAYA, K.M., tekhn.red.

[F.M.IAkovlev, Hero of Socialist Labor] Geroi sotsialisticheskogo truda F.M.IAkovlev. Petrozavodsk, Gos.izd-vo Karel'skoi ASSR, 1959. 38 p. (MIRA 12:11)
(IAkovlev, Fedor Matveevich)

APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6"

"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

SHCHERBAKOV, B. A. (Tula); TERENT'YEV, A. I. (Tula)

Damage to heating boilers during operation on gas. Vod. i san. tekh. no.9:22-25 S '61. (MIRA 14:11)
(Boilers)

APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6"

"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

VINOGRADOV, N.A.; TERENT'YEV, A.I.

Automatic machine for cutting slots. Mashinostroitel' no.9:18-19 S '64. (MIRA 17:10)

APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6"

"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

TERENT'YEV, A.N.

STAVROVSKIY, Ye.L., inzh.; TERENT'YEV, A.N., inzh.

Agricultural machinery at the Leipzig Fair of 1958. Mekh.i elek.sots. sel'khoz. 16 no.5:57-59 '58. (MIRA 11:11)

1. TSentral'naya mashinoispytatel'naya stantsiya (for Stavrovskiy).
2. Vsesoyuznyy nauchno-issledovatel'skiy institut mekhanizatsii sel'skogo khozyaystva (for Terent'ev).
(Leipzig--Agricultural machinery--Exhibitions)

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

RUSINOV, Fedor Mikhaylovich; POPOV, Lev Grigor'yevich; TERENT'YEV, A.N., inzh., retsenzent; PUZRYAKOV, V.A., inzh., red.; NAKHIMSON, V.A., red.izd-va; SMIRNOVA, G.V., tekhn.red.

[Automatic control of tractors and self-propelled agricultural machinery] Avtomatizatsiia upravleniia traktorami i samokhodnymi sel'skokhozaistvennymi mashinami. Moskva, Gos.nauchno-tekhn. izd-vo mashinostroit.lit-ry, 1959. 82 p. (MIRA 13:2)
(Agricultural machinery)

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

L 12971-63 EPF(c)/EWT(j)/EWT(m)/BDS ASD Pc-4/Pr-4 RM/WW
ACCESSION NR: AT3[illegible] S/2513/63/013/000/0003/0007

67

AUTHORS: Luskina, B. M.; Terent'yev, A. N.; Syavtsillo, S. V.

TITLE: Wet oxidation analysis of organosilica compounds containing various elements

SOURCE: AN SSSR. Komissiya po analiticheskoy khimii. Trudy v. 13, 1963. Organicheskiy analiz, 3-7

TOPIC TAGS: H, C, Cl, Si, sulfuric acid, chromic acid, siloxane

ABSTRACT: Organosilica polymers containing metals or nonmetals are very resistant to oxidation or thermal effect. Two types of methods are employed in the analysis of organosilica compounds: The method of dry combustion which is performed in a stream of oxygen with a consequent analysis of H, C, Cl, and Si, and the method of wet oxidation based on the mineralization of the elemento-organic molecule with concentrated acids and additions of various oxidizers or catalysts which in the end permit the analysis of silica and the metal. The proposed method is based on the oxidation of the sample with a mixture of concentrated sulfuric and chromic acids in an oxygen atmosphere at a temperature of 150C in the apparatus shown in the enclosure. The remaining uncombusted

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substance is burned in the quartz tube over chromium oxide absorbed on the surface of pumice. The temperature for phosphor-containing siloxanes was raised from 150C to 180C to obtain a complete decomposition. The organic-silicatitano compounds were decomposed in a mixture of acids at 150C without prior treatment with H_2SO_4. In all cases, carbon was determined gravimetrically silica and sulumina volumetrically, phosphorus and titanium colormetrically and chloride argentometrically. Orig. art. has: 3 tables and 1 figure.

ASSOCIATION: None

SUBMITTED: 00 DATE ACQ: 13Jun63 ENCL: 02

SUB CODE: CH, EL NO REF SOV: 003 OTHER: 000

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10

Ca

A modification of the preparation of *p*-hydroxyphenylaminoacetic acid. A. Terentiev and S. Chernin. *Khim. Farm. Prom.* 1933, 18-19.—*p*-Aminophenol (300 moles) in boiling water is treated with 150 moles of CH_2ClCO_2H, heated and stirred for 1 hr., acidified with HCl, centrifuged and washed with cold H_2O. The product is purified by dissolving in NH_4OH, treating with hyposulfite and filtering through charcoal. The ppt. (with HCl) is washed and dried at 40-5°. L. Nasarevich

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

Polymerization of cyclopentadiene. A. P. Terent'ev and L. A. Solokhin. *Sintet. Kauchuk* 1933, No. 5, p. 12. A study of cyclopentadiene polymerization was made by means of the diazo method for detn. of the C_5H_6 monomer (*C. A.* 28, 3338[7]). The results of expts. of Stobbe and Reuss (*C. A.* 6, 3275), who used the refraction method to measure C_5H_6 polymerization, were compared with the results obtained by the authors. The refraction method showed polymerization about 1.5-2.0% less than reported by Stobbe and Reuss for similar conditions. With the diazo method, however, the indicated polymerization was considerably less. The refraction analysis is based on the reaction $2C_5H_6 \rightarrow C_{10}H_{12}$ and does not take into consideration further polymerization of dimer to polymers, which, it is believed, actually takes place and produces the increased n. James Sorrel

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Determination of cyclopentadiene by reaction with diazo compounds. A. P. Terent'ev and L. A. Sokokhin. *Sintet. Kauchuk* 1933, No. 5, 12-15.—The reaction between C_5H_6 and *p*-nitrobenzenediazonium chloride in AcOH soln. can be used for the detn. of this compd. in mixts. with its polymer. James Sorrel

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ca

Analysis of strong oleum with large samples. A. P. Terent'ev and M. I. Ivanova. *Zavodskaya Lab.* 1933, No. 5, 402; *Chimie & industrie* 31, 1121. To avoid loss by spattering, either of the following methods can be used: (1) Weigh 25-35 g. oleum and add $Na_2SO_4.10H_2O$ (at least 0.4 g. per g. of SO_3, taking care to use crystals that are not too small). Loss of SO_3 can be avoided by the use of a simple app. in which the vapors that are given off in waves are absorbed in water in a volumetric flask. (2) Add H_2O to the sample in such a way that, instead of coming directly into contact with the oleum, it first comes into contact with dil. H_2SO_4, which is gradually strengthened by the oleum until complete mixing is effected. A. Papineau-Couture

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The use of bivalent chromium salts in the determination of organic substances by reduction. A. P. Terent'ev and G. S. Goryacheva. *Uchennie Zapiski* (*Wiss. Ber. Moskau Staats-Univ.*) 3, 277-82(1934); *Chem. Zentr.* 1935, II, 3954.—Examples given are the detns. of *quinone*, *azobenzene* and *m*- and *p*-*nitroaniline* by titration with $CrCl_2$ with neutral red as an indicator. The $CrCl_2$ soln. was prepd. as follows: $K_2Cr_2O_7$ was boiled with HCl until no more Cl_2 was evolved, reduced in the cold with Zn dust and filtered directly into NaOAc. The difficultly sol. brown-red crystals of the acetate pptd. were rapidly washed and dissolved in a 2-3% HCl soln. under an atm. of CO_2. The soln. is standardized against ferriammonium sulfate in the presence of KSCN. W. A. Moore

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B-II-1

BC

Determination of aniline in presence of nitroaniline. A. P. Terentiev and L. A. Schtschetinina (Anilinokras. Prom., 1934, 4, 359—362).—The solution, containing o-, m-, and p-$NH_2 \cdot C_6H_4 \cdot NO_2$ and NH_2Ph, is diazotized at 0°, excess of 30% NaOH is added, and the resultant solution titrated with 0·05N-β-$C_{10}H_7 \cdot OH$, which reacts only with PhN_2Cl. R. T.

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

Determination of *o*-nitroaniline in a mixture with large quantities of *p*-nitroaniline. A. P. Terent'ev, V. A. Khmova and V. P. Puzuireva. *Anilinokrasochnaya Prom.* 4, 554-7(1934).—The detn. is based on the sepn. of the 2 isomers by distn. with steam. Det. the sum of *o*- and *p*-$O_2NC_6H_4NH_2$ by titration with $NaNO_2$. Place 10 g sample and 750 cc. H_2O in a Wurtz flask provided with a dropping funnel and a reflux condenser and boil with a gradual addn. of 200 cc. H_2O in a Babo funnel for 40 min until 450 cc. of the distillate has come over. Wash the condenser with dil. HCl, dil. to 500 cc., and titrate the ortho isomer in the distillate with 0.1 *N* $NaNO_2$ against I and starch soln. The method when checked with prepd. mixts. of pure isomers is accurate to 0.3% with 2.5% and to 0.4% with 7.5% ortho isomer. The method was successfully used in the control of nitration by sapong. and then proceeding as above. The presence of $C_6H_4(NO_2)_2$ has no effect on the results. Because $PhNH_2$ is diazotized much more slowly than $O_2NC_6H_4NH_2$, the detns. can be made in the presence of a little $PhNH_2$. C. B.

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CA

Diazometric methods of determining diene hydrocarbons. A. P. Terent'ev, K. V. Vinogradova and G. D. Hal'pern. *Compt. rend. acad. sci. U. R. S. S.* [N. S.], 4, 267-72(1935).—By titrating the unreacted p-$O_2NC_6H_4N_2Cl$ (I) with β-C_6H_4O, it was possible to det. the amt. of diene hydrocarbon in a mixt. I reacted with only 1,3-diethylenic hydrocarbons. Aromatic hydrocarbons or compds. with conjugated heteroatoms did not react. 2,3-Dimethylbutadiene-1,3, cyclohexadiene-1,3 and cyclopentadiene reacted mol. for mol. with I. This method is applicable to the detn. of divinyls in cracked and pyrolyzed products.

Julius White

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Ca

Detection and determination of naphthalene-1,5-disulfonic acid in the presence of naphthalene-1,6-disulfonic acid. A. P. Terentev and E. M. Terenteva. *Sci. Repts. Moscow State Univ.* 1936, No. 6, 189-205.—$1,5\text{-}C_{10}H_6(SO_3H)_2$ (I) but not $1,6\text{-}C_{10}H_6(SO_3H)_2$ (II) yields a yellow ppt. of $C_{10}H_6Br_6$ when treated with $KBrO_3$-KBr in acid soln.; the reaction will serve to detect not less than 0.01% of I. With II partial substitution of Br takes place but no insol. product is formed. To det either I or II treat the aq. soln. of the K salt with 30% of Na-Hg and after 14 hrs. titrate the SO_3^{--} formed with I. With I both SO_3H groups are eliminated but with II only 1 SO_3H group. **Detection and determination of naphthalene-1-sulfonic acid in the presence of naphthalene-2-sulfonic acid.** A. P. Tarentev and K. K. Magaram. *Ibid* 207. 11.—$1\text{-}C_{10}H_7SO_3K$ (III) but not $2\text{-}C_{10}H_7SO_3K$ (IV) yields $C_{10}H_7Br$ when treated with $KBrO_3$-KBr; not less than 0.0125% of III suffices to give the test. Treatment of III but not IV with Na-Hg (as above) gives SO_2 which can be detd. iodometrically. In this way III can be detd. alone or in presence of IV. B. C. A.

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1 3

BC

Influence of reaction conditions on the yields of isomerides in nitration of acetanilide. A. P. TERENTIEV and B. M. KEDROV (Sci. Rep. Moscow State Univ., 1936, No. 6, 213—234).—The content of o-$N(I)_2 \cdot C_6H_4 \cdot NH_2$ (I) in the product of nitration of NHPhAc rises from 6—7% when 100% H_2SO_4 is taken to 28% with 84% H_2SO_4; nitration does not proceed when the H_2SO_4 contains >16% of H_2O, whilst the use of 10% oleum leads to production of tarry products. Increasing the amount of 100% H_2SO_4 taken per g. of NHPhAc from 2 to 5 ml. greatly lowers the yield of (I), but further addition of H_2SO_4 does not further reduce it. The yield of (I) is slightly increased by raising the nitration temp. from −3° to 10°, whilst further rise to 40° has no effect. The m-$NO_2 \cdot C_6H_4 \cdot NH_2$ content of the product is independent of temp., concn. and amount of H_2SO_4 taken. Addition of AcOH or H_2SO_4 does not affect the relative yields of (I) and p-$NO_2 \cdot C_6H_4 \cdot NH_2$. R. T.

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B-II-1

DIAZOMETRIC DETERMINATION OF DIENE HYDROCARBONS. II. DETERMINATION OF INDIVIDUAL DIENE HYDROCARBONS. III. DETERMINATION OF DIENE HYDROCARBONS IN PYROBENZINES. Ap. D. Terentiev, E. V. Vinogradova, and G. D. Galpern (Sci. Rep. Moscow State Univ., 1936, No. 6, 243-247, 249-255).

II. $(CH_2 : CMe)_2$ or α-cyclohexadiene, in presence of C_6H_{14}, C_7H_{16}, C_6H_6, cyclo-hexane and -hexene are determined by the method ofoTerentiev et al. (A., 1936, 744).

III. Conjugated dienes in cracking benzines are determined by the above method.

R.T.

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7-3

Action of diazo-compounds on unsaturated compounds. Determination of mono- and polymeride of phenyllestadiene. A. P. TERENTIEV and M. E. ZEGELMAN (Sci. Rep. Moscow State Univ., 1936, No. 6, 257—261).—$CHPh:CH\cdot CH:CH_2$ (I), but not its dimeride, combines with diazotised p-$NO_2\cdot C_6H_4\cdot NH_2$. The reaction of polymerisation is one of the second order. C_5H_5N reacts with (I), and is not a suitable solvent for studying velocity of polymerisation. R. T.

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CA

Diazometric analysis of dienes and its applications. A. P. Terent'ev. *Org. Chem. Ind.* (U. S. S. R.) 4, 535-42(1937).—The method is applicable to detn. of cyclopentadiene, $(CH_2CMe)_2$, $CHPh:CHCH:CH_2$, $CH:Me:CHCMe:CH_2$, $CH_2:CClCH:CH_2$ and pyrrole. B. C. P. A.

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

The action of aromatic diazo compounds on unsaturated compounds. I. A. P. Terent'ev. *J. Gen. Chem.* (U. S. S. R.) 7, 2026–7 (1937).—T.'s work on the detn. of dienes by addn. of diazo compds. is reviewed. II. **A sensitive reaction for bivinyl.** A. P. Terent'ev and E. M. Ivanova. *Ibid.* 2028–9.—When p-$C_6H_4(NH_2)_2$ in H_3PO_4 (d. 1.7) is diazotized at −10° by $NaNO_2$ in concd. H_2SO_4, a reagent is obtained which gives a red to red-brown color with hydrocarbons contg. conjugated double bonds. This reaction is very sensitive with cyclopentadiene and biisopropenyl, but less so with bivinyl. However, if gas contg. bivinyl is passed through a solvent and the soln. treated with this reagent, as little as 0.2 vol. % of bivinyl can be detected. H. M. Leicester

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CA

Two reactions for detecting cyclopentadiene. A. P. Terent'ev and M. I. Ivanova. *J. Gen. Chem.* (U. S. S. R.) 7, 2087-91(1937). The color test of Afanas'ev (*C. A.* 30, 1330[2]) is not specific for cyclopentadiene (I). When gas contg. at least 0.25 mg. of I is passed through an aq. soln. of $Hg(NO_3)_2$ slightly acidified with HNO_3, a white cloudiness appears. C_6H_6, MePh, C_2H_4, bivinyl and isoprene do not interfere but butenes and C_2H_2 also cause ppts., though not as rapidly. When 12-15 cc. of gas contg. 0.05% of I is passed into a 0.25% soln. of *p*-benzoquinone in EtOH, a deep-blue color develops. Even butene and C_2H_2 do not interfere. The color is probably due to intermediate addn. compds. similar to those formed in the reaction of Kesting (*C. A.* 23, 4941).

H. M. Leicester

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PROCESSES AND PROPERTIES INDEX

The action of aromatic diazonium compounds on unsaturated compounds. III. A. P. Terent'ev and A. A. Demidova. *J. Gen. Chem.* (U. S. S. R.) 7, 2564-70 (1937); cf. *C. A.* 32, 4817.—Biallyl does not couple with diazotized p-$NO_2C_6H_4NH_2$ (I) or 2,4-$(NO_2)_2C_6H_3NH_2$ (II), while 2,4-hexadiene couples with both. The product with II m. 127-9° (decompn.). This confirms the fact that Me groups in conjugated systems favor coupling. 1,3-Pentadiene reacts with diazotized p-$C_6H_4(NH_2)_2$ (III) and II and more slowly with I. The product with II m. 165-7° (decompn.). 2,5-Dimethyl-2,4-hexadiene reacts rapidly with III, more slowly with II to give a product m. 148-51°, and very slowly with I. Isobutylene reacts with II and III but not with I. Trimethylethylene reacts slowly with III and with II to give a product m. 176-7°. These are the 1st monoölefins to react with diazonium compds. Me groups at the end of chains promote coupling more than when they are within the chain.

H. M. Leicester

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CN

10

The action of aromatic diazonium compounds on unsaturated compounds. IV. Unsaturated cyclic and aromatic hydrocarbons. A. P. Terent'ev and L. L. Gomberg. *J. Gen. Chem.* (U. S. S. R.) 8, 662-7 (in English 667-8) (1938); cf. *C. A.* 32, 2004[8].—Styrene, dimethylstyrene, fluorene, phenanthrene and anthracene do not couple with diazotized p-$O_2NC_6H_4NH_2$ (I) and 2,4-$(NO_2)_2C_6H_3NH_2$ (II), while cyclopentadiene couples easily with both. Indene reacts slowly with II and not at all with I. The product with II m. 110-12° (decompn.). The results show that Ph radicals at the ethylene bond inhibit coupling.

Chas. Blanc

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Cleaning the benzene scrubbers with steam. ... Terent'ev and Ya. B. Chertkov. *Coke and Chem.* (U. S. S. R.) 1959, No. 11, 47; *Khim. Referat. Zhur.* 2, No. 4, 115(1959).—Results of cleaning the scrubbers with steam in the Staro-Makeev coke-chem. plant are given. Remove the oil from the scrubber, leave an opening of approx. 2 in. in the cover to create a partial vacuum, connect the scrubbers with a common collector for draining oil and condensate and force steam for 18-20 hrs. under a pressure of 5 atm. at 170° through 2-in. pipes. Up to 50 tons of steam is used per scrubber. The condensate seps. easily into layers without addnl. steam. The upper layer of oil is reused. Steaming is recommended twice a year.

W. R. Henn

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Ca

Lecture aids in stereochemistry. A. P. Terent'ev
Uspekhi Khim. 9, 948-9(1940). Hints to instructors.
F. H. Rathmann

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Ca

Determination of active hydrogen with the Grignard reagent in an atmosphere of carbon dioxide. A. P. Terentyev and K. D. Shcherbakova. *Zhur. Obshch. Khim.* (J. Gen. Chem. R.) 10, 2041-6(1940). A method for det'g active H in an atm. of CO_2 was developed. The CO_2 enters the reaction vessel (after complete air replacement) under a layer of ethereal soln. of the substance to be analyzed, to eliminate any initial reaction between CO_2 and CH_3MgI (I). The stream of CO_2 is now broken. The methane formed replaces the CO_2 from the vessel and the reaction is completed in an atm. of methane. Dry CO_2, which is now passed through, carries over the methane into the azotometer and reacts with the excess of I. By transferring the gas from the azotometer into the eudiometer it is measured, as it is often done in Dumas's N det'n. The method has many advantages. It is unnecessary to bring the app. to the initial temp.; it eliminates the use of a gas buret with large quantities of Hg; it permits the use of a wide selection of solvents and particularly the use of abs. ether. The reaction can be carried out at a higher temp., driving off the ether. Under exptl. conditions, especially with primary amines, Mg org. compds. were very reactive. James J. Lichtin

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1ST AND 2ND ORDERS

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3RD AND 4TH ORDERS

ca

2

Minimal and equilibrium concentration. I. A. M. Belousov and A. P. Terent'eva. *J. Applied Chem.* (U. S. S. R.) 13, 463-9(in French, 469)(1940).—The effect of conditions and medium upon the relation between a min. concn. and the mass-action law was investigated in the case of the reaction of $BaSO_4$ formation. The coeff. of excess, β, representing the relation between min. and equil. concns., and the coeff. of decrease of sensibility $\tau = 1/\beta$ were introduced for observing an effect of the medium on the min. concn. In most cases β had a value of the order 10^4. The following formula is recommended for the detn. of min. concn.: $C_{min.} = \beta(L_p/C_1)$, where L_p is the soly. product, C_1 is concn. of reacting ion, and β a const. equal (in the first approximation) to 1×10^4. 15 references.

A. A. Podgorny

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FERENTIEV, A.I.

BC

a-3

Transaction with the use of acrylonitrile. II. Synthesis of β-N-substituted propionitriles. A. Y. Terentiev and E. A. Terentieva (J. Gen. Chem. Russ., 1942, 12, 415—417).—Although the general addition reaction of primary and sec. amines at the C:C of α-β-unsaturated nitriles is well known, few details are available for the case of $CH_2:CH\cdot CN$. The reaction between $CH_2:CH\cdot CN$ and piperidine is studied, and a new synthesis of αγ-dipiperidinopropane is described.

β-Piperidinopropionitrile (I) is prepared by adding $CH_2:CH\cdot CN$ (21·8 g.) to piperidine (34 g.) in a flask fitted with a reflux condenser; the reaction is spontaneously exothermic. The product is then heated for 4 hr. (temp. not stated), giving (I) (~80 %), b.p. 100—102°/5 mm., d_4^{20} 0·9437, n_D^{20} 1·4712, MR_D 50·33 (calc. 46·00). It gives a sparingly sol. *platinichloride*, $C_8H_{16}N_2Cl_2Pt$, and a nitrate, m.p. 157·5—158°. Reduction to γ-piperidino-α-propylamine (II) is carried out by treating (I) (30 g.) [illegible] boiling EtOH (560 ml.) with Na (40 g.). Addition of H_2O [illegible] of the aq. solution 3 times with Et_2O gives (II) [illegible]/10 mm., d_4^{20} 0·8964, n_D^{20} 1·4720, MR_D 44·38 (calc. 44·[illegible]), [illegible] a very strong base; it gives a picrate, m.p. 205—206°, but no insol. compound with $PtCl_4$. With $[CH_2]_3Br_2$ (22 g.) in C_6H_6 (50 ml.) under reflux for 4 hr. (II) (12 g.) gives (after treatment with conc. caustic alkali) αγ-dipiperidinopropane (yield not stated), b.p. 141—142°/10 mm., d_4^{20} 0·9153, n_D^{20} 1·4668, MR_D 66·17 (calc. 66·03). It gives a sparingly sol. *platinichloride* and a picrate, m.p. 190°. G. R. S.

Lab. Org. Chem. im N. D. Zelinskiy, Moscow State U.

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Ca

10

Syntheses with acrylonitrile. I. A laboratory method of preparation and the properties of acrylonitrile. A. P. Terent'ev and R. V. Vinogradova (Moscow State Univ.). *J. Gen. Chem.* (U.S.S.R.) 14, 1044-6(1944); cf. *C.A.* 37, 3995[4].—Acrylonitrile (I) is prepd. conveniently in the lab. as follows. First a soln. of 600 g. KCN in 700 cc. water is added during 15-20 min. with cooling to a soln. of 1140 g. $MgSO_4.7H_2O$ in 2 l. of water in a 5 l. thick-walled flask. Cooled ethylene oxide is then added in a thin stream with shaking and continued cooling. After stoppering tightly, the flask is shaken at intervals and cooling is continued. After 15-30 min., the reaction begins, as is indicated by pptn. of $Mg(OH)_2$. If necessary, cracked ice may be added to the mixt. to keep the reaction under control. After standing 10-12 hrs., the mixt. is neutralized with 50% H_2SO_4 under the hood. Pptd. sulfates are filtered off and washed with EtOAc. The filtrate is concd. by evapn. on the water bath, the cryst. ppt. filtered off and washed with EtOAc. Next the aq. filtrate is extd. several times (7-10) with EtOAc and the combined EtOAc exts. distd., first under atm. and finally under reduced pressure, to obtain β-hydroxypropionitrile (II) in 85-90% yield based on the KCN. Dehydration of II to I is effected by heating 50-100-g. portions of II with 10% by wt. of powd. Sn, which is prepd. by first treating a concd. aq. soln. of $SnCl_2$ with Zn dust, then treating the pptd. Sn with AcOH to dissolve unreacted Zn, filtering, washing with alc., and drying. The dehydration is carried out simultaneously with distn. of the water and I formed thereby in 75-85% yield. Crude I is dried with potash and redistd. to obtain pure I, b. 78°, d_4^{20} 0.811, n_D^{20} 1.3900. J. W. Perry

Lab. Org. Chem. im. Zelinsky, Moscow State U.

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CA

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Determination of active hydrogen with the Grignard reagent in an atmosphere of carbon dioxide. II. A. P. Terent'ev and K. D. Shcherbakova (Lab. Organicheskoi Khimii im. N. D. Zelinskogo Moskov. Gosudarst. Univ.). *J. Gen. Chem.* (U.S.S.R.) 15, 86-9(1945); cf. C.A. 35, 4308[7].—Various alcs., glycols, phenols, acids, and amino derivs. were analyzed by the method previously described. The method yielded good results with all the above compds., except tertiary alcs. and compds. insol. in ether. The former gave higher results, presumably because water was split off in the presence of MeMgI. The latter do not react with MeMgI because of their insoly. in Et_2O.

A. A. Podgorny

Translation - CTS 44, 26 Jun 53

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Ca

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Isomerization of the dimethyl ether of 1,4-dihydro-α-naphthohydroquinone. A. P. Terent'ev and P. P. Shavalova (Org. Chem. Lab. of Moscow State Univ.), *J. Gen. Chem.* (U.S.S.R.) 18, 142-5(1948)(English summary).—It was shown that the di-Me ether of 1,4-dihydro-α-naphthohydroquinone (5,8-dihydro-1,4-naphthalenediol) when heated with NaOMe isomerizes into the corresponding 1,2-dihydro deriv. The initial 1,4-dihydro-α-naphthoquinone was prepd. by condensation of butadiene with quinone and the product treated with HBr in AcOH gave *5,8-dihydroxy-1,4-dihydronaphthalene*, m. 212°, which, treated with Me_2SO_4 in 20% NaOH, gave the *di-Me ether* (I) of *1,4-dihydro-α-naphthohydroquinone* m. 50°, b. 298-300°. The ether heated with 10% MeONa in MeOH to 130° for 6 hrs. in a sealed tube isomerized quantitatively into the *di-Me ether* (II) of *1,2-dihydro-α-naphthohydroquinone*, m. 54°. I yields a cryst. *dibromide* (2,3-dibromo-1,2,3,4-tetrahydro-5,8-dimethoxynaphthalene), m. 124°; II fails to yield a stable dibromide. Heating of either ether with S at 250° yielded the mono-Me ether of naphthohydroquinone, m. 84-6°. II shows an *MR* exaltation of 2.34; I has a normal *MR*. Reduction of II in MeOH by means of Na gave the Me ether of tetrahydronaphthohydroquinone, m. 39-40°; I failed to reduce under these conditions. II copolymerizes readily with butadiene (Na catalyst); I does so very slowly. G. M. Kosolapoff

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CA

7

Determination of active hydrogen by the Grignard reagent in carbon dioxide atmosphere. III. A. P. Lyubeniev and K. D. Shcherbakova (Moscow State Univ.). J. Gen. Chem. (U.S.S.R.) 16, 855-8(1940); cf. C.A. 40, 1420[3].—It was shown that active H can be detd. by the new method in acids, amines, and phenols with the following solvents: C_6H_6, xylene, $CHCl_3$, and $(CH_3Cl)_2$. In all cases MeMgI was used as the reagent. G. M. K.

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Syntheses by means of acrylonitrile. III. Synthesis of 1-diethylamino-3-aminopropane. A. P. Terent'ev and A. N. Kost (Moscow State Univ.). *J. Gen. Chem.* (U.S.S.R.) 16, 859 (2:1946); cf. *C.A.* 37, 3095[1].—CH_2CHCN (26.5 g.) was added to 40 g. Et_2NH, and, after the initial heat evolution, was heated on a steam bath 2 hrs. to yield 80–95% *3-diethylaminopropionitrile*, b_{12} 65°, b_{17} 76°, b_{20} 87°, b_{11} 112°, b_{111} 197°, d_4^{20} 0.8761, n_D^{20} 1.5480; *HCl salt* m. 120° (from water); *picrate* m. 85° (from MeOH); *methiodide* m. 152° (from MeOH); *ethiodide* m. 168° (from MeOH); *phosgene deriv.* m. 124° (from $CHCl_3$). Hydrolysis by boiling concd. HCl 12 hrs. or 25% NaOH 5 hrs. gave *3-diethylaminopropanoic acid*. The amino nitrile (25 g.) and 150 cc. BuOH were treated with 30 g. Na in large lumps; after completion of the reaction (2 hrs.) the mixt. was treated with 100 cc. water, acidified with HCl, and steam distd. On addn. of NaOH to the residue and extn. with Et_2O there was obtained 58–63% *1-diethylamino-3-aminopropane*, b_{12} 61.2°, b_{14} 85.7°, b_{14} 99–100°, b_{17} 168–70°, d_4^{20} 1.4435. G. M. Kosolapoff

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TERENT'YEV, A. P.

"Furance Sulfonation," Dok. AN, 51, No. 9, 1946. "Sulphuration of Indoal," Dok. AN, 51, No. 9, 1946. State Univ. Lomonosov, Moscow, 1945-. (S. K. Golubeva)

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Catalytic Dismutation of Hydro-Aromatic Ketones. A. P. Terentev and A. N. Guseva. 10 pages. Battelle translation from *Reports of the Academy of Sciences of U.S.S.R.*, v. 52, 1946, p. 135-138.

Gives results of experimental work on the application of "irreversible catalysis" to the dismutation of cyclohexanone, 1-methyl-cyclohexanone 2, and cyclopentanone. Cyclohexanone yields a mixture of mostly phenol and cyclohexanol; methyl-cyclohexanone yielded mostly the corresponding compounds; cyclopentane did not react similarly. In both the first two cases, it was found possible to reverse the reaction to some extent.

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The stereoisomerism of 2-pyrrolecarboxaldoxime. A. P. Terent'ev and A. N. Makarova. *Vestnik Moskov. Univ.* 1947, No. 4, 97-100.—On oximation of 2-pyrrolecarboxaldehyde (0.3 g.) with $NH_2OH.HCl$ *without heating* wherein 2 kinds of crystals were obtained: 0.2 g. of a fine cryst. powder m. 164° (I) (from $CHCl_3$), identical with the oxime of Bamberger and Djerdjan (*Ber.* 33, 536 (1900)), and 0.1 g. of coarser crystals (II) m. 70-1°, which, when heated above the m.p., first fused to a clear melt, became turbid at 120-130°, solidified at 150-160°, and melted again at 164°; I and II evidently are stereoisomers. The unstable II is converted into I by recrystn. from hot water or from alc., by treatment with HCl, or by irradiation in benzene with a quartz Hg lamp; in air, II goes over into I in 2-3 weeks. By analogy with other aldoximes, II is the syn form. With $Cu(OAc)_2$, I gave no ppt. in dil. soln., only a color change from blue to green; in concd. soln. a green Cu salt pptd., sol. in dil. acids and in NH_3, and contg. 1 atom Cu per mol. oxime; II gave a brown ppt. even in very dil. soln., insol. in dil. acids and in NH_3, and contg. approx. 1/2 atom Cu per mol. oxime.

N. Thon

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CA

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The stereoisomerism of the oxime of 2-acetylpyrrole. A. P. Terent'ev and A. N. Makarova. *Vestnik Moskov. Univ.* 1947, No. 4, 101–4.—Short (1.5–2 hrs.) boiling of 2-acetylpyrrole in alc. with $NH_2OH.HCl$ gave only one oxime, m. 145°. On *long* (10 hrs.) boiling, the product m. 90° and can be sepd. by crystn. into a compd. m. 82° (I) and the ordinary oxime m. 145° (II). I is a stereoisomer of II which can be converted into I by heating in soln., by melting to 160°, or by distn.; conversion of I into II can be brought about by the action of ZnIMe, by passing a stream of HCl through an ether soln., or by 30 hrs. irradiation in benzene with a quartz Hg lamp; I is the more thermostable form. Beckmann rearrangement gave with I and with II the same product, identified as *N*-methyl-2-pyrrolecarboxamide, m. 151°; evidently, I was converted into II under the action of traces of HCl; while not conclusive, this reaction would indicate that II is the anti form. With a satd. aq. soln. of $Cu(OAc)_2$, the ether soln. of I gives a brown ppt., insol. in dil. AcOH, sol. in glacial AcOH and in pyridine, contg. 0.5 atom Cu per mol. oxime, and I is evidently *syn*-Me 2-pyrryl ketoxime. II gives a ppt. only in concd. soln.; the salt is a typical electrolyte, dissolves in H_2O with a green color and in NH_4 with a bright-blue color, and contains 1 atom Cu per 1 oxime; II evidently is *anti*-Me 2-pyrryl ketoxime. N. Thon

Translation CTS, 44, 26 Jun 53

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Determination of active hydrogen by Grignard reagents in a carbon dioxide atmosphere. IV. A. P. Terent'ev, K. D. Shcherbakova, and N. V. Kremenskaya (Moscow State Univ.). *J. Gen. Chem.* (U.S.S.R.) 17, 100-4 (1947)(in Russian); cf. *C.A.* 41, 1575g. The use of MeMgBr and MeMgCl, in place of the usual MeMgI, in the active H detn. in CO_2 atm. as described previously was investigated. MeMgBr is suitable on the basis of trials with the following substances: BuOH, borneol, pyrocatechol, MeBtC(OH)Ph, $Ph_2C(OH)Et$, PhCH(OH)Et, BzOH, o-$AcOC_6H_4CO_2H$, mandelic acid, citraconic acid, $PhNH_2$, p-$MeC_6H_4NH_2$, 1-(1-piperidyl)-3-aminopropane, HCONHPh, and 2-acetylpyrrole. The deviations noted were: RtPhCHOH gave 1.11 and Et_2Ph_2COH 1.25 H atoms, evidently because of the dehydrating action of the reagent. Primary amines gave figures slightly lower than theoretical ($PhNH_2$ 1.94, 1-(1-piperidyl)-3-aminopropane 1.8 H) due to interaction of CO_2 with the primary reaction product, so that the 2nd stage does not go quite to completion for the less sol. bromides, while the sol. iodides are able to go to completion. MeMgCl gave consistently good results with: 1- and 2-naphthols, salicylic acid, borneol, p-nitrophenol. Hydroquinone, because of poor solv., gave low results (1.56-1.63). 2-$C_6H_7NH_2$ gave 0.99 H at Et_2O reflux, and 1.98 H at 100°. Benzidine gave 2.0 H at low and high (100°) temps. $AcCH_2CO_2Et$ gave 1.01 H. When the reaction was run in CdI_2, thymol gave 1.03 H, salicylic acid 2.0, and Ph_2NH 1.0 H. Generally, MeMgCl was less active than the Br compd. and tended to lose its titer more rapidly than the latter on standing.

G. M. Kosolapoff

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Syntheses with acrylonitrile. IV. Reduction of nitriles with sodium in alcohol medium. A. N. Kost and A. P. Terent'ev (Moscow State Univ.). *J. Gen. Chem.* (U.S.S.R.) 17, 105-8(1947)(in Russian); cf. *C.A.* 41, 1005h.—In unpublished work of Longinov and Filippov (1933) it was found that when the Na used for the reduction of esters contains even as little as 0.01% of K the yields of the alcs. drop rapidly. In the reduction of $BuCH(CO_2Et)_2$ the yield is zero when the Na contains 0.1% K. However, if the amt. of K is increased, the yields rise again and become normal with 2% K. This is important since Na is usually contaminated with traces of K. This study was extended to the reduction of $Et_2NCH_2CH_2CN$ and 1-piperidinepropionitrile in BuOH by the method of Suter and Moffett (*C.A.* 28, 1650[1]). Tech. Na gave 38-50% of the diamine, pure Na 51-63%, while tech. Na with the addn. of 2% K gave 65.9-70% diamine, in the case of $Et_2NCH_2CH_2CN$. With the piperidyl compd. the yields were 42-46%, 48.9-57.2%, and 48-57%, resp., thus indicating a similarity to the results obtained in ester reduction. The high-K Na gave reproducible yields and tended to give smaller amts. of higher-boiling products (presumably secondary amines). The Rupe technique (immediate steam distn.) gave similar results, but was inconvenient because of the low volatility of the products. G. M. K.

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264. **Determination of Active Hydrogen by Means of the Grignard Reagent in an Atmosphere of Carbon Dioxide. V. Determination of Moisture in Technical Products.** (In Russian.) A. P. Terent'ev, D. G. Kadaner, and Iu. K. Kopchenova. *Journal of General Chemistry* (U.S.S.R.), v. 17(79), May 1947, p. 913-916.

According to the English abstract of this article, it is shown that the method of Terentiev and Shcherbakova may be successfully used for the determination of moisture in various substances and technical products that do not react with methylmagnesium iodide. Examples of the determination of moisture in charcoal, clay, starch, and benzene are reported. However, pages 914 and 915 are missing from the copy received at Battelle.

Lab. Org. Chem,
Moscow State U.

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CA

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Syntheses with acrylonitrile. V. Preparation of urethans of 1-diethylamino-3-aminopropane. A. P. Terent'ev and A. N. Kost (Moscow State Univ.). *J. Gen. Chem.* (U.S.S.R.) 17, 1185-8(1947)(in Russian); cf. *C.A.* 41, 1609h.—$Et_2N(CH_2)_3NH_2$ ($^1/_{10}$ mole) in 30 cc. Et_2O was treated with $^1/_{10}$ mole powd. K_2CO_3 followed by water until a paste formed (about 1.5 cc.). The mixt., cooled with ice, was treated slowly with $^1/_{10}$ mole RO_2CCl (or generally RO_2CCl) in 30 cc. Et_2O; after standing at room temp. 15-20 hrs. the org. layer was sepd., the aq. layer extd. with Et_2O and the org. solns. distd. *in vacuo*. The following *esters of (3-diethylaminopropyl)carbamic acid (1-diethylamino-3-propane urethans)* were prepd. in this way: *Et* (66.7%), b_{10} 130°, n_D^{20} 1.4503; *Pr* (53.2%), b_{10} 123.3°, n_D^{20} 1.4452, n_D^{25} 1.4403, d_4^{20} 0.9307; *Bu* (42.5%), b_{10} 132°, n_D^{20} 1.4513, d_4^{20} 0.9311; *octyl* (63.3%), b_5 181.5-2°, n_D^{20} 1.4528, n_D^{25} 1.4477, d_4^{20} 0.9168; *cyclohexyl* (46.5%), b_{10} 165-7°, n_D^{20} 1.4725, n_D^{25} 1.4752, d_4^{20} 0.9765; *2-methylcyclohexyl* (81.5%), b_5 177°, n_D^{20} 1.4603, n_D^{25} 1.4723, d_4^{20} 0.9670; *menthyl* (88.2%), b_5 173°, n_D^{20} 1.4710, d_4^{20} 0.9483, crystd. on standing and m. 31° (from MeOH). G. M. Kosolapoff

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Syntheses with acrylonitrile. VI. Preparation of 1-dimethylamino-3-aminopropane. A. P. Terent'ev and A. N. Kost (Moscow State Univ.). J. Gen. Chem. (U.S.S.R.) 17, 1632-6(1947) (in Russian); cf. C.A. 42, 1566i.—Tech. Me_2NH or Me_3NH sulfate in 50% aq. soln. was poured in a thin stream onto solid NaOH, and the Me_2NH so generated was passed over soda lime, then through a series of absorption flasks contg. 106 g. dry CH_3CHCN, cooled with snow; absorption was complete in 6-8 hrs., and after an addnl. 20 min. passage of the amine, the solns. were allowed to stand 2 hrs. and were distd. to give 80-1% β-dimethylaminopropionitrile, bis 168-70°; on redistn., it bis 171°, d_4^{20} 0.8705, n_D^{20} 1.4282; picrate, m. 151° (from MeOH); HCl salt, m. 199° (from MeOH); methiodide, m. 153° (from MeOH); ethiodide, m. 128.5° (from MeOH); ethobromide, m. 157° (from MeOH-Et_2O); propobromide, m. 189° (from MeOH-Et_2O); allochloride, m. 185-7° (from MeOH). The nitrile (20 g.) in 450 cc. dry BuOH was rapidly added to 30 g. Na contg. 2% K and the mixt. was immediately heated so that the soln. was complete in 20-5 min. (use of pure Na triples the reaction time). On cooling, 100 g. H_2O was added and the BuOH distd. with steam, which also removed the diamine; a calcd. amt. of 2 N HCl was used in the receiver. The distillate was slowly concd. with a stream of steam and auxiliary heating to remove the BuOH and most of the H_2O; the residue, treated with solid NaOH, extd. with Et_2O, and the ext. dried over NaOH, gave 52-6% 1-dimethylamino-3-aminopropane (I), bis 70-80°; redistn. gave the pure diamine, bis 44-5°, bis 133°, d_4^{20} 0.8272, n_D^{20} 1.4415; HCl salt, m. 184° (from MeOH); picrate, m. 211° (from EtOH). If the reduction is done with pure Na there is formed 1-2.2 g. higher-boiling fraction, from which can be isolated 0.4 g. bis(3-dimethylaminopropyl)amine, bis 128-31°, n_D^{20} 1.4531; HCl salt, hygroscopic flakes (from Et_2O-HCl); picrate, m. 200° (from water); chloroplatinate, orange cubes. I (3.1 g.) in 30 cc. Et_2O and 4.1 g. K_2CO_3 were treated with 1.5 cc. water, followed by 3.3 g. $ClCO_2Et$ in 30 cc. Et_2O added slowly with cooling; after stirring 8 hrs. and standing overnight, distn. of the org. soln. gave 55.8% Et 3-dimethylaminopropylcarbamate, bis 137-8°, d_4^{20} 0.9653, n_D^{20} 1.4480. 1-Menthyl chloroformate gave 51.8% 1-menthyl analog, bi 164.5°, d_4^{20} 0.9557, n_D^{20} 1.4700; on standing 5 yrs. this solidified and m. 45° (from dil. MeOH).

G. M. Kosolapoff

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

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*212. **Determination of Active Hydrogen by Means of Grignard Reagent in a Carbon Dioxide Atmosphere. VI.** (In Russian.) A. P. Terent'ev and N. I. Shchor. *Zhurnal Obshchei Khimii* (Journal of General Chemistry), v. 17(79), Nov. 1947, p. 2075-2079.

Describes a series of modifications of the method previously proposed by the senior author and coworkers. It is shown that an ether solution of zinc methyliodide may be substituted for magnesium methyliodide.

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Sulfonation of pyrrole. A. P. Terent'ev and M. A. Shadkhina (Lomonosov State Univ., Moscow). *Compt. rend. acad. sci. U.R.S.S.* 55, 227-9(1947)(in English). - Pyrrole (1 g.), 6.6 g. pyridine sulfotrioxide, $C_5H_5N \cdot SO_3$ (cf. *C.A.* 41, 2066*a*), and 5 ml. CH_2ClCH_2Cl are heated 10 hrs. in a sealed tube on the H_2O bath. The product is heated 1 hr. with excess $Ba(OH)_2$, and the excess Ba is pptd. with CO_2 and filtered hot. The filtrate is concd. to 5-6 ml. and pptd. with alc. to give 3 g. *Ba 3-pyrrolesulfonate*, rendered analytically pure by repeated reppin. from H_2O with alc. The free acid is hygroscopic and darkens rapidly in the air. The *Na salt* is stable but the *Ag salt* darkens rapidly. Oxidation of the Ba salt with $K_2Cr_2O_7$ and H_2SO_4 gives maleimide; hydrolysis with 10% HCl at 90-98° gives no $BaSO_4$; treatment with Br-H_2O yielded dibromomaleimide and $BaSO_4$; boiling with 2% Na-Hg in aq. soln. pptd. $BaSO_3$ and volatilized a base assumed to be "4-aminobutynol"(?); reaction with diazotized p-$NO_2C_6H_4NH_2$ in cold AcOH gives a red color and ppt. of Ba sulfonate (?) mixed with an azo dye, the azo group apparently replacing the sulfo group.

R. W. Fleming

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Sulfonation of furan derivatives. A. P. Terent'ev and L. A. Kazitsyna (Lomonosov State Univ., Moscow). *Compt. rend. acad. sci. U.R.S.S.* 55, 625-8(1947); cf. C.A. 41, 2888a. Silvan (I), 2,5-dimethylfuran (II), coumarone (III), and thiophene (IV) were sulfonated with $C_5H_5N \cdot SO_3$ in 1:3 molar ratio at 100-10° 8-10 hrs. in a sealed tube. I gave 80% 2-methyl-3,5-furandisulfonic acid (V), isolated as the Ba and Na salts. Pure V was not obtained since it is very hygroscopic. II gave 2,5-dimethyl-3-furansulfonic acid as the Ba salt. III gave in quant. yield 2-coumaronesulfonic acid, isolated as the Ba and Ag salts. IV gave a disulfonic acid, the constitution of which has not yet been established. C_6H_6, MePh, and 1,3,5-$Me_3C_6H_3$ were not sulfonated by $C_5H_5N \cdot SO_3$ whereas PhOMe gave a quant. yield of a monosulfonic acid.

W. S. Port

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Sulfonation of indole and its homologs. A. P. Terent'ev and L. V. Tsymbal (Lomonosov State Univ., Moscow). *Compt. rend. acad. sci. U.R.S.S.* 55, 833-5(1947); cf. *C.A.* 41, 2033c.—The Ba salt of *1-indolesulfonic acid* (I) was prepd. by shaking a finely divided aq. suspension of 1 g. indole (II) with 8 g. $Ba(OH)_2$ and 4 g. $C_8H_6N\text{-}SO_3$ in an ice bath 1.5 hrs. The excess $Ba(OH)_2$ was pptd. with CO_2 and removed by filtration. The filtrate was concd. to a small vol. in the const. presence of NH_3 and 1 g. I was pptd. with excess EtOH. Its constitution was proven by analysis and the hydrolysis of I to II. The yield of the Ba salt of *2-indolesulfonic acid* was improved to practically quant. by sulfonation of II with a 2- or 3-molar excess of $C_8H_6N\text{-}SO_3$ at 120-40° (sealed tube). Its constitution was proven by showing that sulfonation takes place in skatole but not in 2-methylindole. The prepn. of $C_8H_6N\text{-}SO_3$ is described.

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LA 10

Sulfonation of 2-acetylpyrrole. A. P. Terent'ev and V. A. Yanovskaya (Moscow State Univ.). *Vestnik Moskov. Univ.* 3, No. 10, 155-7(1948). See C.A. 44, 3073.
G. M. Kosolapoff

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CA

Sulfonation and sulfonic acids of acidophobic substances. X. Sulfonation of 2-acetylfuran. A. P. Terentiev, L. A. Kantsyna, and A. M. Turovskaya (Moscow State Univ.). *Vestnik Moskov. Univ.* 3, 159-60(1948).

Zhur. Obshchei Khim. (J. Gen. Chem.) 20, 185-7(1950); cf. C.A. 44, 3973c.—2-Acetylfuran (2 g.) and 5.8 g. pyridine·SO_3 in 10 ml. $(ClCH_2)_2$ after 10 hrs. at 110°, followed by treatment with $BaCO_3$, gave 0.8 g. unreacted acetylfuran and 2.5 g. (82.5%) *Ba 2-acetyl-5-furansulfonate* (from dil. EtOH), which yields $BaSO_4$ in the cold with Br water. The use of more sulfonating agent gave lower yields. Attempted sulfonation with H_2SO_4 and SO_3 in $(CH_2Cl)_2$ gave only tars and no sulfonic acids.

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TKHENT'YAN, P. V.

"In Memory of D. N. Kashkarov, "Priroda, No. 5, 1948.

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TERENT'YEV, A. P.

Terent'yev, A. P. - "Sulfonation of N substituted pyrroles," Vestnik Most. un-ta, 1948, No. 11, p. 123-31

So: U-3566, 15 March 53, (Letopis 'Zhurnal 'nykh Statey, No. 13, 1949)

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TERENT'YEV, A. P.

Terent'yev, A. P. "The subdual of sulfuric acid (on new sulfur compounds)," Vestnik vyssh. shkoly, 1948, No. 12, p. 46-51

SO: U-3264, 10 April 1953, (Letopis 'Zhurnal 'nykh Statey, no. 3, 1949)

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Sulfonation and sulfamic acids of acidophobic compounds. I. Sulfonation of furan. A. P. Terent'ev and L. A. Kaltsyna. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 18, 723-8(1948); cf. *C.A.* 42, 5966.—Pyridine-SO_3 was found to be the most suitable sulfonating agent for furan. The reagent, prepd. according to Baumgarten (*C.A.* 19, 817; 20, 3309), m. 175° (decompn.). Tech. furan was purified through the maleic anhydride adduct, which was decompd. at 140-50°; pure furan, b. 31.2-1.5°, n_D^{20} 1.4234, d_4^{20} 0.9392. Synthetic furan, prepd. in 9.6% yield by decarboxylation of pyromucic acid by heating with Ph_2NH, b. 31.4-2°, n_D^{20} 1.4236, d_4^{20} 0.9390. The sulfonations were conducted by heating to 100° 8-10 hrs. in sealed tubes, treating the mixt. with an aq. paste of $BaCO_3$ on the steam bath 30-40 min., filtering hot, evapg., and pptg. *Ba furansulfonate* with EtOH. In typical expts. 1.9 g. furan gave a 30% yield when 4.4 g. pyridine-SO_3 was used, 56% with 8.8 g., and 90% with 13.4 g. The Ba salt was purified by pptn. from H_2O by EtOH; with Na_2CO_3 it gave the *Na salt*, purified as above. The Ba salt gives with *S-benzylthiuronium chloride* a difficultly sol. *salt*, decomp. 205°. The Ba sulfonate (1 g.) treated with the theoretical amt. of H_2SO_4 and filtered, gave on evapn. the *free furansulfonic acid* as an oily liquid, which deposits crystals rapidly deliquescing in air. Oxidation of the Ba salt with Br water gave Ba maleate dihydrate and $BaSO_4$, thus indicating that sulfonation proceeds in the α-position. The furansulfonic acid is stable to hot alkalies but is decompd. by boiling 20-5% HCl or H_2SO_4, yielding S oxide and the corresponding HO deriv. G. M. K.

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Synthesis with acrylonitrile. VII. *N-Ethyl-N-phenyl-β-aminopropionitrile* and some of its derivatives. *I. P. Terent'ev, A. N. Kost, and V. M. Potapov (Moscow State Univ.). J. Gen. Chem. (U.S.S.R.) 18, 82-6(1948)(in Russian); cf. C.A. 42, 2578d.*—In a systematic study of the best reaction conditions for $CH_2:CHCN$ (I) and PhNEtH interaction, it was found that the best yields were attained with an excess of I, the mixt. being heated 100 hrs. on a steam bath in the presence of Ac_2O and hydroquinone. Higher temps. lead to tar; Fe or Ni salts almost do not affect the yield, while Cu powder actually lowers it; in the presence of EtONa and in the absence of a catalyst the reaction does not proceed at all; use of a little H_2SO_4 at 180° gives a very poor yield. HCl is not a satisfactory catalyst as it gives volatile N salts which are difficult to remove. Passage of I and PhNEtH over Al_2O_3 or glass wool at 200–400° failed to produce any reaction. Freshly distd. PhNEtH (60.5 g.), 31.8 g. I, and 1 g. Ac_2O were heated, in the presence of a trace of hydroquinone, in a sealed tube 100 hrs. on a steam bath, giving 70% *N-ethyl-N-phenyl-β-aminopropionitrile* (II), b_4 151–60°; on redistn., it b_4 158°, b_{10} 164–5°, freezes to a glass at −40°, n_D^{20} 1.5503, d_4^{20} 1.0260; *HCl salt*, hygroscopic mass; the *picrate* and *styphnate* are oils; the *coupling product* with diazotized *sulfanilic acid* is a green solid giving a yellow-green aq. soln., turning to brown on acidification; the *coupling product* with diazotized *p-$H_2NC_6H_4VO_3$* is red-brown, insol. in water; *tetrazotized benzidine* couples very slowly in acid soln., giving a red-violet soln. which turns yellow in neutral or alk. soln. II (14 g.), 20 g. KOH, 20 ml. H_2O, and 70 ml. EtOH were refluxed 15 hrs., acidified by HCl, and extd. with iso-BuOH; addn. of Et_2O to the ext. gave *N-ethyl-N-phenyl-β-aminopropionic acid-HCl*, the filtrate evapd. in vacuo, gave 12.5 g. *β-phthalimidopropionitrile*, m. 183.4° (from EtOH). This (5 g.) refluxed with 40 cc. 20% HCl 6 hrs., cooled, filtered (almost 100% recovery of $C_6H_4(CO)_2NH$ results), the filtrate evapd. in vacuo at 60°, the dry residue extd. with hot abs. EtOH, the cooled ext. filtered, the filtrate again evapd. in vacuo, and the residue dissolved, in 6 parts of 90% EtOH, filtered, and evapd. at 65°, gave 3 g. *β-alanine-HCl*, m. 116–18°, almost free of inorg. impurities. Phthalimide (14.7 g.) heated to 80° was treated with 0.05–0.01 g. hydroquinone and 5 cc. of Me_2PhNOH soln., the stirred mixt. treated over 10–15 min. with 50 cc. Me acrylate and a few crystals of hydroquinone, simultaneously with 10–15 cc. of Me_2PhNOH soln., heated to 80–95° 20 mins. after complete soln. took place, filtered hot, and evapd. at 40–60°; the residual oil solidified on standing, to *1,2-$C_6H_4(CO)_2N$-$CH_2CH_2CO_2Me$*, m. 65–77°, 91–3% yield; the pure product, m. 73–5° (from EtOH, then from benzene). Use of Et acrylate similarly gave the corresponding *Et ester of phthaloyl-β-alanine*, m. 70–2°. The Me ester (35 g.) and 370 cc. 20% HCl boiled 9 hrs., cooled, filtered, and the filtrate evapd. on a steam bath and dried in a desiccator, yielded 79–83% *β-alanine-HCl*, m. 116–20° (from 90% EtOH). Free *β-alanine* was obtained by mixing 10 g. of the HCl salt with 30 g. Pb oxide and 50 cc. H_2O, boiling 3 min., evapg. to dryness, drying the residue 3 hrs. at 60–80°, extg. with hot H_2O, treating the ext. with H_2S, evapg., and drying over $CaCl_2$ to give 5.15 g. (72.3%) *β-alanine* (m.p. of successive fractions: 193–4.5°, 196.5–8°, 197–8°). This (0.5 g.) in a little H_2O treated hot with an excess of fresh hydrated CuO, boiled several min., filtered, and concd. gave the blue Cu salt of alanine as the hexahydrate. Heating 4 g. *β-alanine* and 7 g. phthalic anhydride to 170° 15 min. gave *phthaloyl-β-*

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of II, m. 194–5° (decompn.); this results also from III and EtCOCl with 3-methylpyridine in 34% yield. III and HzH give 64% of the benzylidene analog, m. 166°. II (1 g.), refluxed 1 hr. with 50 ml. H_2O, gives 93% $AcCH_2NHBz$, m. 84° (*2,4-dinitrophenylhydrazone*, golden, m. 214–15°). I (1 g.), refluxed 2 hrs. with 100 ml. abs. EtOH, gives a quant. yield of $AcCH(CO_2Et)NHBz$, b. 150°/10^{-4} mm. (5–10% reconversion to I) (*semicarbazone*, m. 172–3° (decompn., not cor.)). $MeCH(NHBz)CO_2Na$ (5 g.) and 7.5 ml. Ac_2O, heated 5 min. at 100°, give 1.5 g. 2-phenyl-4-methyl-5(4*H*)-oxazolone and 1 g. *Me 1-benzamidoethyl ketone*, pale yellow, b. 110–30° (*2,4-dinitrophenylhydrazone*, pale yellow, m. 199–200° (not cor.)); in a 2nd expt. (heating 1 min. at 100°), there resulted a compd. $C_{11}H_{12}O_4N_2$, m. 225–6° (decompn., not cor.). II could not be reduced in aq. soln. with Pt oxide or small quantities of Raney Ni. II (2 g.) in 20 ml. 0.5 *N* NaOH and 4 g. Raney Ni, shaken 20 hrs. with H at room temp. and atm. pressure (4 mols. H absorbed), give 54.5% $MeCH(OH)CH(NH_2)CO_2H$ (V), m. 238–9°; microbiol. assay showed that this contained about 7% DL-threonine (VI). II (10 g.) in 110 ml. 0.5 *N* NaOH, hydrogenated over 20 g. Raney Ni until 1 atom H was absorbed, gave 6 g. unchanged II and 2.6 g. (63% on basis of II consumed) DL-*N-benzylallothreonine*, m. 238° (decompn.); further reduction in *N* NaOH over Raney Ni (12 hrs.) gave 70% V. II (20 g.) in 200 ml. 0.5 *N* NaOH, hydrogenated (2 hrs.) over 20 g. Raney Ni at 85–90°/110 atm. and the crude product (7.5 g.) ustd. with 15 ml. H_2O, give, on adds. of 100 ml. hot alc. to the filtrate, 3 g. V; the insol. portion (4.2 g.), crystd., repeatedly from AcOH, yields *α-[(cyclohexylmethyl)amino]-β-hydroxybutyric acid* (VII), m. 231–2°; reduction at 100–10°/125 atm. gives only VII. VII (1 g.), 7 ml. HI, and 0.17 g. red P, heated 6 hrs. at 100°, give 65% *α-[(cyclohexylmethyl)amino]butyric acid*, m. 300–2° (decompn. and sublimation); *HCl salt*, m. 211–13° (decompn.); *Et ester-HCl*, m. 118–20°; the acid was prepd. also in 67% yield from (cyclohexylmethyl)amine and $EtCHBrCO_2H$. $AcCH(CO_2Et)NHBz$ (220 g.) and 800 ml. abs. EtOH, refluxed 1 hr., 15 g. Raney Ni added, the refluxing continued 30 min., the filtrate hydrogenated (1–3 hrs.) over 50 g. Raney Ni at 100°/75–100 atm., and the crude HO ester (97%) hydrolyzed with HBr, gives 64% V (on basis of hippuric acid); the ester b. 110°/10^{-4} mm. and partially crystd., the crystals being used to seed the crude ester. The ester (265 g.) in 1.1 l. ether, allowed to stand 3 days at 0°, gives 104 g. (38% based on II) DL-*N-benzoylallothreonine Et ester* (VIII), m. 98.5–101.5°; mild alk. hydrolysis gives 93% DL-*N-benzoylallothreonine* (IX), m. 177°. The ethereal filtrate yields 108 g. of a yellow oil, which yields 66 g. (18% on basis of II) of the $PhCH_2CH_2NH_2$ salt (m. 161–2°), decompn. of which gives 13% DL-*N*-benzoylthreonine (on basis of II) and (with HBr) about 12% of DL-threonine. VIII (240 g.) in 850 ml. dry C_5H_5N at −10°, treated with 192 g. *p*-$MeC_6H_4SO_2Cl$ (3.5 hrs.) and poured into 1.2 l. concd. HCl and 2.4 kg. ice, gives 276 g. DL-*N-benzoyl-O-(p-tosyl)-allothreonine Et ester* (X), m. 83.5–3° (from petr. ether) or 148.5–51.5° (from AcOEt-petr. ether); 276 g. crude X, 138 g. AcOK, and 950 ml. anhyd. EtOH, refluxed 2.5 hrs., give 36% (on basis of II) of DL-*trans-4-carbethoxy-2-phenyl-5-methyl-Δ²-oxazoline* (XI), $MeCH.CH(CO_2Et).N:CPh.O$, b. 100°/10^{-4} mm.; *picrate*, m. 127.3°; *HCl salt*, m. 117.5–18°; 150 g. XI and 750 ml. 23% HBr, refluxed 4 hrs. and the sirupy residue dissolved in 750 ml. hot EtOH and a slight excess of concd. NH_4OH, give 56 g. (26% on basis of II) of VI. VIII (20 g.) and 50 ml. $SOCl_2$, kept 1 hr. at room temp., give 31% (based on II) of a mixt. of *cis*- and *trans*-XI; the HCl salt, on standing in the air, absorbs H_2O and gives DL-*O-benzylthreonine Et ester-HCl*, m. 167°; *picrate*, bright yellow, m. 140.5°. DL-Allothreonine has no effect on the growth of *Strep. faecalis* in the presence or absence of VI. NH_4Br, a possible contaminant of crude samples of the amino acid, also is without effect on the growth of the organism. The total over-all yield of VI from hippuric acid is about 30%, the greatest loss being incurred during the sepn. of the HO ester.

C. J. West

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USSR/Chemistry - Propionitrile, Beta (N-Piperidyl) Mar 1948

Chemistry - Synthesis

"Synthesis With the Aid of Nitrile Acrylic Acid. VIII. 1-(1'-Piperidyl)-3-Amino-Propane and Its Urethan Derivatives," A. P. Terent'yev, A. N. Kost, Lab Org Chem imeni Academician N. D. Zelinskiy, Moscow State U, 5 pp

"Zhur Obshch Khim" Vol XVIII (LXXX), No 3

Piperidine and acrylonitrile were used to produce beta-(N-piperidyl)-propionitrile. Latter was reduced by sodium potassium alloys to produce 1-(1'-piperidyl)-3-amino-propane. Article describes

69T79

USSR/Chemistry - Propionitrile, Beta (N-Piperidyl) (Contd) Mar 1948

properties of some eight new compounds. Submitted 24 Feb 1947.

69T79

TERENT'YEV, A. P.

APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6"

"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

TERENT'YEV, A. P. PA 8/49T46

USSR/Chemistry - Acids, Sulfo Apr 48
Chemistry - Sulfuration

"Sulfuration and Sulfo Acid in Acidophobic Compounds," A. P. Terent'yev, L. A. Kazitsyna, Lab Org Chem imeni N. D. Zelinskiy, Moscow State U, 5 3/4 pp

"Zhur Obshch Khim" Vol XVIII (LXXX), No 4

Describe unique sulfurizing agent for acidophobes - pyridine - sulfotrioxide. Furanmonosulfo acid was prepared for the first time. Some of its salts were also isolated. Submitted 8 Mar 1947.

[redacted] 8/49T46

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

CA

Simplified preparation of diethyl diacetylsuccinate. A. P. Terent'ev and L. A. Yanovskaya. *Zhur. Obshchei Khim.* (J. Gen. Chem.) **18**, 1159-60(1948).—Powd. Mg (6.7 g.), activated by warming with a little iodine, is added to 250 g. $AcCH_2CO_2Et$, stirring continued until the reaction is complete (2-5 hrs.), and the Mg deriv. filtered from the ester excess and washed with $(CH_2Cl)_2$; the recovery of unreacted ester averages 185 g. The Mg deriv., suspended in ClC_2H_4, is treated, with stirring, with 38 g. Br added dropwise in 2 vols. $C_2H_4Cl_2$ with cooling (in the initial stages); after filtration, the $MgBr_2$ ppt. is washed with the same solvent, the soln. freed of solvent by distn., followed by blowing with steam, and the liquid layer of *di-Et diacetylsuccinate* washed with Na_2CO_3 for conversion to the solid form, which is filtered off and laid; yield, 85%. The procedure is much more satisfactory than the customary Na process. G. M. K.

Lab. Org. Chem,
Moscow State U.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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USSR/Biology
Photography

Jul/Aug 48

"Photography of Small Objects With the FED Camera," A. P. Terent'yev, V. A. Shpanauer, 5 pp

"Byul Mosk Obshch Ispytat Prirod, Otdel Biol" Vol LIII, No 4

Describes new "Fokkad" transparent focusing and framing cylinder which is combined with a lens attachment to simplify considerably the photographing of various objects on a 1:1 or 1:2 scale. The "Fokkad" is of simple construction, and is expected to greatly expand the applications of the FED camera in zoology,

60/49T8

USSR/Biology (Contd)

Jul/Aug 48

botany, and other sciences. Shows several examples of the camera's capabilities, with sketches of the camera and attachments.

60/49T8

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A. P. TERENT'YEV

A. P. Terent'yev and A. N. Kost

"A Method of Preparing the Dinitril of Succinic Acid," in Yezhemesyachnyy Byulleten' Izobreteniy, (Monthly Bulletin of Inventions), No. 12, 1949, of the Patent Administration, Committee on Inventions and Discoveries, Council of Ministers USSR. The patents were granted on 31 December 1949.

Dinitril of succinic acid is obtained by reacting acrylic acid nitril with an alkali metal cyanide. The reaction is carried out in water at 0-30° in the presence of magnesium sulfate. The succinic acid dinitril is isolated by the known method of extracting with butyl acetate.

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TERENT'YEV, A. P.

62/49T14

USSR/Chemistry - Sulfonation
Chemistry - Pyrrole

Mar 49

"Sulfonation and Sulfoacids of Acidophobic Compounds. III, Sulfonation of Pyrrole and Its Homologues," A. P. Terent'yev, L. A. Yanovskaya, Lab of Org Chem, Moscow State U, 5 1/2 pp

"Zhur Obshch Khim" Vol XIX, No 3

Prepared pyrrole-2-sulfonic acid, 1-methylpyrrole-2-sulfonic acid, 2-methylpyrrole-5-sulfonic acid, 2,4-dimethylpyrrole-5-sulfonic acid, and 1-o-tolylpyrrole-2-sulfonic acid by sulfonation of pyrrole and its homologues by pyridine-sulfur

62/49T14

USSR/Chemistry - Sulfonation (Contd)

Mar 49

trioxide in a dichloroethane solution. Even at 150° C, 2,5-dimethylpyrrole was not sulfonated. Submitted 3 Nov 47.

62/49T14

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

TERENT'YEV, A. P.

FA 65/49T36

USSR/Chemistry - Indole
Sulfonation

Apr 49

"Sulfonating and Sulfo Acids of Acidophobic Compounds: IV, Sulfonating Indole and Its Homologues," A. P. Terent'yev, S. K. Golubeva, L. V. Tsymbal, Lab of Org Chem, Moscow State U, 3 pp

"Zhur Obshch Khim" Vol XIX, No 4

Sulfonated indole and skatole by heating to 120° with pyridinesulfotrioxide, and obtained barium salts as well as indolsulfonic-(2) acid and 3-methylindolsulfonic-(2) acid. 2-Methylindole is not sulfonated even at 170°. Processing the

65/49T36

USSR/Chemistry - Indole (Contd)

Apr 49

indole in a water medium in the presence of baryta produced the salt of indolsulfonic-(1) acid, which was readily hydrolyzable in an alkaline solution. Submitted 3 Nov 47.

65/49T36

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

TERENT'YEV, A. P. PA 65/49T37

USSR/Chemistry - Ethers, Vinyl Sulfonation Apr 49

"Sulfonating and Sulfo Acids of Acidophobic Compounds: V, Sulfonating Vinyl Ethers," A. P. Terent'yev, N. P. Volynskiy, Lab of Org Chem, Moscow State U, 2½ pp

"Zhur Obshch Khim" Vol XIX, No 4

During the activity of pyridine-sulfotrioxide on simple ethers of vinyl alcohol, compounds of two molecules of sulfur anhydride with double bonds are produced. During the acid hydrolysis of the barium salt of the derived acid, sulfoacetic aldehyde is formed. Submitted 10 Nov 47.

65/49T37

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

USSR/Chemistry - Sulfonation Jul 49
Pyrrole

"Sulfonation Sulfo Acids of Acids phobic Compounds: VI, Sulfonation of Alpha-Alpha'-Substituted Homologues of Pyrrole," A. P. Terent'yev, L. A. Yanovskaya, Lab of Org Chem, Moscow State U imeni M. V. Lomonosov, 4 pp

"Zhur Obshch Khim" Vol XIX, No 7

Sulfonated 2,5-dimethylpyrrole, 1, 2, 5-trimethylpyrrole, and 2, 3, 5-trimethylpyrrole with pyridinesulfotrioxide. Then, prepared barium salts of: 2,5-dimethylpyrrolesulfonic-(3) acid, 2, 5-dimethylpyrroledisulfonic-(3,4) acid, 1,2,5-trimethylpyrrole-

2/50T63

USSR/Chemistry - Sulfonation Jul 49
Pyrrole (Contd)

sulfonic-(3) acid, 1,2,5-trimethylpyrroledisulfonic-(3,4) acid, and 2,3,5-trimethylpyrrolesulfonic-(4) acid. Submitted 21 Mar 48.

2/50T63

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

PA 149T28

USSR/Chemistry - Sulfonation Aug 49
Acidophobic Compounds

"Sulfonation and the Sulfonic Acids of Acidophobic Compounds: VII. Sulfonation of Unsaturated Hydrocarbons," A. P. Terent'yev, A. V. Dombrovskiy, Lab of Org Chem, Moscow State U, 4½ pp

"Zhur Obshch Khim" Vol XIX, No 8

Pyridinesulfotrioxide does not enter into reaction (up to 150° C) with paraffins, cycloparaffins, homologues of benzene, and olefins with double bonds not adjacent to the end carbon

149T28

USSR/Chemistry - Sulfonation (Contd) Aug 49

atom. However, alpha-olefins (hexene-1, heptene-1) react at 150° C. Prepared barium salts of sulfonic acids of cyclohexene, methylenecyclohexane, camphene, styrene, and indene. Submitted 7 May 48.

TERENT'YEV, A. P.

149T28

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CA

Sulfonation and sulfonic acids of acidophobic compounds. VIII. The investigation of sulfonation agents

A. P. Terent'ev, L. A. Kazitsyna, and S. E. Suvorova (Moscow State Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 19, 1951-4(1949); cf. *C.A.* 43, 214e; 44, 1481i. H_2SO_4 yields only tars with furan derivs. regardless of the solvents used; reaction with sylvan is similar. In the presence of Me_3NPh or pyridine with 1 mole H_2SO_4, no reaction takes place even at 120°, while higher proportions of H_2SO_4 give tar. SO_3 gave tars and only traces of sulfonic acids with furan or sylvan even in Ac_2O soln.; sulfonation by pyridine-SO_3 succeeds only when equimolar amts. of reagents are used, as excess of pyridine hinders the reaction; traces of SO_3 act similarly. $Me_3N.SO_3$ (obtained by direct interaction) gives up its SO_3 sluggishly and yields but 15% fransulfonic acid in 20 hrs. at 100°. Treatment of furan with dioxane-SO_3 gave tars and S-contg. acids of products of ring opening of furan and dioxane. Picoline.-SO_3 behaves analogously to the previously described pyridine analog for sulfonation reactions. G. M. K.

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

Sulfonation and sulfonic acids of acidophobic substances. IX. Sulfonation of 1- and 2-acetylpyrrole. A. P. Terent'ev and L. A. Yanovskaya (Moscow State Univ.). *Zhur. Obshchei Khim.* (*J. Gen. Chem.*) 19, 2118-22(1949); cf. *C.A.* 44, 1095i, 1954h.—2-Acetylpyrrole (I) (1.3 g.) in 25 ml. 6% oleum, poured into H_2O, neutralized with $BaCO_3$, filtered, and concd. gave 75% *Ba 2-acetyl-4-pyrrolesulfonate*, sol. in H_2O, giving $BaSO_4$ with Br water, and SO_2 on boiling with 15% HCl, while oxidation with CrO_3 gave sulfonaleamic acid, isolated as the Ba salt, $BaC_4H_4O_5SN$. I (1.00 g.) and 4.77 g. pyridine-SO_3 in $(CH_2Cl)_2$ in 8 hrs. at 100°, then 3 hrs. at 120-30°, give on evapn. and neutralization with $BaCO_3$, 1.2 g. *di-Ba 2-acetyl-3,5-pyrroledisulfonate* (from dil. EtOH), while the mother liquor yields the monosulfonate described above (0.7 g.). 1-Acetylpyrrole (1.5 g.) and 5 g. pyridine-SO_3 in $(CH_2Cl)_2$ in 5 hrs. at 100° and 6 hrs. at 110-20° give 2 *disulfonates*, apparently the *2,5-* and *2,4-isomers*, isolated as the *Ba salts* by fractional pptn. from dil. EtOH; oxidation by CrO_3 gave *N*-acetylmaleimide, m. *54°*.

G. M. Kosolapoff

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

Sulphonation of α-phenylbutadiene. A. P. Terentyev and A. V. Dombrovskii (*C. R. Acad. Sci., U.R.S.S.*, 1949, [illegible], 313–318).—1-Phenylbuta-1:3-diene is sulphonated at $C_{(4)}$ with $C_5H_5N{\cdot}SO_3$; oxidation affords BzOH and H_2SO_4. 1-Phenylbuta-1:3-diene-4-sulphonic acid is reduced (Raney Ni) by controlled reduction to 1-phenylbut-3-ene-4-sulphonic acid which is oxidised to dihydrocinnamic acid, thus proving the constitution.

CHPh:CH·CH:CH_2 dissolved in $(CH_2Cl)_2$ stabilised with *m*-$C_6H_4(NO_2)_2$ is heated at 100° for 4 hr. with $C_5H_5N{\cdot}SO_3$. The cooled mass is poured into dil. aq. NH_3, the solvent is removed, and 1-phenylbuta-1:3-diene-4-sulphonic acid is isolated as the *Ba* salt, converted by aq. Na_2CO_3 into the *Na* salt (I). The latter is oxidised by 1% aq. $KMnO_4$ to BzOH and H_2SO_4, the odour of PhCHO can be detected at the beginning of oxidation. I is totally reduced by H_2-Raney Ni in H_2O at room temp. but oxidation by aq. $KMnO_4$ when half the H_2 has been taken up affords CHPh·CH_2·CO_2H.

H. Taubkin.

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"APPROVED FOR RELEASE: 07/16/2001 CIA-RDP86-00513R001755320020-6

TERENT'YEV, A. P.

PA 66/49T23

USSR/Chemistry - Sulfonation Aug 49
Dienes

"Sulfonating Diene Hydrocarbons as a Displacement Reaction," A. P. Terent'yev, A. V. Dombrovskiy, Moscow State U imeni M. V. Lomonosov, 3¼ pp

"Dok Ak Nauk SSSR" Vol LXVII, No 5

Conducted experiments with divinyl, isoprene, diisopropenyl, alphaphenylbutadiene, and cyclopentadiene, heated to 80-120° C in a solution of dichloroethane with double molecular quantities of pyridine-sulfotrioxide and then processed with ammonia and baryta. The barium salt was separated with alcohol. Submitted 1 Jun 49.

66/49T23

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Reversible changes of the pH of solutions of some organic compounds on illumination. A. P. Terent'ev. *Doklady Akad. Nauk S.S.S.R.* **68**, 537-9(1949). On exposure to a quartz Hg lamp the following changes of pH of acid solns. were observed: AcOH (2%) + 0.10, succinic (2%) + 0.10, diphenic (satd. soln.) + 0.10, salicylic (satd. soln.) + 0.10, lactic (2-3%) + 0.30, aminoacetic (5%) + 0.21, anthranilic + 0.20, benzoic 0, *o*-chlorobenzoic 0, citric (10%) + 0.25, tetrachlorophthalic 0, sulfanilic 0, benzenesulfonic 0, fumaric −0.10, tartaric (5%) −0.10. Only with the last-named acid was the change irreversible on screening. Reversibility in the case of fumaric acid indicates that the change of pH is not an isomerization. With bases the changes were: NH_4OH (5%) + 0.30, ethylenediamine (4%) + 0.25, propylenediamine (4%) + 0.30, pyridine (50%) 0. All changes were reversible. With alcs. the changes were: MeOH (50%) + 0.40, EtOH (50%) + 0.45, iso-AmOH (satd. soln.) + 0.30, PhOH (2%) 0, *α*-naphthol + 0.23, resorcinol + 0.26, mannitol (10%) + 0.35, sorbitol (10%) + 0.30, glucose (20%) + 0.20, picric acid + 0.30, dioxane (50%) 0, acetone (50%) + 0.24. On the whole, the effect is absent in mols. in which rotatory changes of configuration are unlikely. Distd. H_2O (pH = 7.2 at 25°) and solns. of HCl or H_2SO_4 (pH 1.3-2.0) showed no changes on illumination. N. Thon

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Cyanoethylation of acetone. A. P. Terent'ev and S. M. Gurvich. *Vestnik Moskov. Univ.* 5, No. 5, *Ser. Fiz.-Mat. i Estest. Nauk* No. 3, 47-51(1950); cf. *C.A.* 42, 4912h.—To 470 g. boiling Me_2CO contg. 4 g. NaOH was added with stirring over 1 hr. 212 g. $CH_2:CHCN$; after refluxing 2 hrs., the mixt. was acidified with HCl and the org. layer gave on distn. approx. 80% undistillable solid residue as well as 35 g. $AcCH_2CH_2CH_2CN$, b_6 93-8°, b_{760} 238-40°, d_4^{20} 0.9747, n_D^{20} 1.4330 (*semicarbazone*, m. 131° (from EtOH)), and 48.5 g. $AcCH(CH_2CH_2CN)_2$, b_6 190-6°, d_4^{20} 1.0185, n_D^{20} 1.4680; the distn. residue on crystn. from aq. MeOH gave about 24% $AcC(CH_2CH_2CN)_3$, m. 152° (cf. Bruson and Riener, *C.A.* 37, 1379[9]). Reduction of $AcCH_2CH_2CH_2CN$ with Na and BuOH gave *2-methylpiperidine*, b_{760} 118-19°, d_4^{20} 0.8412, n_D^{20} 1.4430; *picrate*, m. 164° (from H_2O); [Lipp, *Ann.* 289, 210(1896), gives m. 134-5°, while Marckwald, *Ber.* 29, 43(1896), gives m. 127-8°]; *HCl salt*, m. 207° (from dioxane); *oxalate*, m. 135° (from EtOH-Et_2O). The higher fraction of the reduction products yielded *6-amino-2-hexanol*, b_6 98-100°, d_4^{20} 0.9365, n_D^{20} 1.4702, *HCl salt*, hygroscopic solid; *picrate* and *flavianate*, liquids; *oxalate*, m. 100° (from EtOH-Et_2O); *1-naphthylurethane*, m. 107° (from aq. MeOH). G. M. Kosolapoff

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Synthesis of 1 oxa 5 azacyclooctane A. N. Kost, A. P. Terent'ev, and V. G. Yashunskii. *Vestnik Moskov. Univ. Ser. 6, No. 6, Ser. Fiz.-Mat. i Estest. Nauk* No. 4, 41-2(1950). — Condensation of $CH_2:CHCN$ and $HOCH_2CH_2CN$ with aq. KOH catalyst gave 88.7% $O(CH_2CH_2CN)_2$. This, reduced with Na and BuOH, upon prolonged steam distn., acidification of the distillate, evapn., and treatment with solid NaOH, gave 32.8% cyclic base, $C_6H_{13}NO$, b_{760} 167.8°, b_{16} 60.2°, d_4^{20} 0.8532, n_D^{20} 1.4288, which contained approx. the correct amt. of active H required by $O(CH_2CH_2CH_2NH$. The product has high affinity for CO_2 and fumes in air. Its *HCl salt* is very hygroscopic; *picrate*, m. 96°; *1-naphthylurea*, m. 117-18°; *chloroplatinate*, decomp. 119° (from H_2O); the *styphnate* and *benzenesulfonamide deriv.* are oils. The amt. of NH_3 liberated during the reduction approximates the yield of the cyclic amine (12.5% of total N of the diamine).

G. M. Kosolapoff

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Action of organomagnesium compounds on acrylonitrile

A. P. Terent'ev and A. N. Kost. [illegible] Nauk No. 5, 41 [illegible]. Addn. of MeMgI, EtMgBr, BuMgCl, PhMgBr, or PhCH$_2$MgCl in Et$_2$O at room temp. to CH$_2$:CHCN causes spontaneous polymerization of anionoid type of the latter; a 100% yield of ppt. forms even at −10° almost instantaneously. The polymer, after washing and pptn. from pyridine by H$_2$O, is a light yellow solid, sol. in pyridine, iso-AmOAc, and MeNO$_2$, softening at 160–80°. Rast mol. wt. about 900. Metallic Na does not react with CH$_2$:CHCN even after 100 hrs. at 90°. G. M. Kosolapoff

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Salicylalamines in analysis. I. Production and properties of salicylalamines. A. P. Terent'ev and E. G. Rukhadze (Moscow State Univ.) *Zhur. Anal. Khim.* 5, 211-16(1950). The synthesis of 7 salicylal monoamines and 4 diamines from an alc. soln. of salicylaldehyde and an aq. soln. of the corresponding amine is reviewed. The salicylalamines gave qual. tests with Cu^{++}, Ni^{++}, Co^{++}, Pd^{++}, Pt^{++}, Cd^{++}, Zn^{++}, Hg^{++}, and Pb^{++}. They formed either a colored ppt. or a colored sol. complex. Most favorable conditions for the reaction were heating and pH 7-9. Many of H_2O-insol. ppts. dissolved in pyridine, $CHCl_3$, iso-AmOH, AmOH, or Et acetate forming a colored soln.

M. Hosch

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Priority of A. A. Kolli in the establishment of the structure of glucose. A. P. Terent'ev and S. M. Gurvich. S. Thon. *Uspekhi Khim.* 19, 128-33(1950).

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Substitution rules in the pyrrole series. A. P. Terent'ev and L. A. Yanovskaya. *Uspekhi Khim.* 19, 202-19 (1950). – A crit. review; 114 references. N. Thon.

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

B C

A2
6

[illegible] and sulphonic acids of acidophobic compounds. X. [illegible]. A. P. Terentyev, A. A. Kasitsyna, and [illegible] USSR, 1950, 80, 173–176 [U.S. transl. [illegible]] [illegible], m.p. 29.8°, n_D^{20} 1.5012, obtained by [illegible] of Ac_2O with furan in C_6H_6 is sulphonated with C_6H_5[illegible] at [illegible] for 10–12 hr. in $C_6H_6Cl_2$ to yield 2-acetyl-[illegible] acid [illegible] isolated as Be salt, $C_{10}H_{10}O_{10}S_2Be$. [illegible] indicating that the SO_3M [illegible] BRITISH ABSTRACTS.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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Sulfonation and sulfonic acids of acidophobic substances. XI. Application of the leaching method to the investigation of sulfonation products of pyrrole. A. P. Terent'ev, L. A. Yanovskaya, and V. G. Yashunskii (Moscow State Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) **20**, 510-13(1950); cf. C.A. **43**, 7015c, 44, 5862i. Sulfonation of *pyrrole* yielded only the *2-sulfonic acid*. The Ba salt was converted to the Na salt by Na_2CO_3 and this was rubbed with PCl_5, fused, extd. with cold CCl_4, and washed with H_2O; evapn. of the org. soln. gave the *sulfonyl chloride*, which failed to yield the amide with NH_4OH but did yield the *anilide*, m. 142° (from C_6H_6), on treatment with $PhNH_2$. The Ba salt was taken up in aq. MeOH and titrated, with exclusion of CO_2, with 0.1 *N* Na_2CO_3 and phenolphthalein; the results checked $(C_4H_4O_3NS)_2Ba$. The salt (1.627 g.) shaken 2 hrs. with 35 ml. aq. MeOH (2:3 by vol.) gave a satd. soln. at 26° and an aliquot of the soln. was again analyzed, while the aliquot vol. was replaced by an equal vol. of soln. of the same compn., and the process was repeated 4 times; the soly. of the solid phase, i.e., the compn. of the undissolved salt, remained the same. The same procedure used on the unpurified Ba salt obtained by pyridine-SO_3 sulfonation of pyrrole gave a similarly const. result, thus showing the homogeneity of the sulfonation reaction product.

G. M. Kosolapoff

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B A

AII-1

Synthesis using acrylonitrile (vinyl cyanide). IX. Reaction of acrylonitrile with ammonia, and the preparation of trimethylenediamine. A. P. Terentyev, K. I. Churina, and A. N. Kost (*J. gen. Chem. USSR*, 1950, 20, 1073—1078 [U.S. transl., 1115—1120]). — A study is made of the reaction of alkyl cyanides with NH_3 under the conditions used by Whitmore *et al.* (A., 1944, II, 290), and by Ford and Bue (A., 1948, II, 181; 1947, II, 382). With vinyl cyanide the following equilibria occur: $NH_3 + CH_2{:}CH{\cdot}CN \rightleftharpoons NH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CN \rightleftharpoons NH(CH_2{\cdot}CH_2{\cdot}CN)_2 + NH_3 \rightleftharpoons N(CH_2{\cdot}CH_2{\cdot}CN)_3 + NH_3$. The following reactions take place with considerably more difficulty: $NH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CN + CH_2{:}CH{\cdot}CN \rightleftharpoons NH(CH_2{\cdot}CH_2{\cdot}CN)_2$; $NH(CH_2{\cdot}CH_2{\cdot}CN)_2 + CH_2{:}CH{\cdot}CN \rightleftharpoons N(CH_2{\cdot}CH_2{\cdot}CN)_3$. At a low concn. of NH_3 the chief reaction product is the *terf.* amino-cyanide, whilst raising the concn. of NH_3 increases the yield of the primary and *sec.* amino-cyanides. For the polymerisation of $NH_2{\cdot}(CH_2)_2{\cdot}CN$ results opposite to those of Whitmore (*loc. cit.*) are stated; polymerisation occurs without the evolution of NH_3, giving a polymer which is H_2O-soluble, unlike those of $CH_2{:}CH{\cdot}CN$. A laboratory method for the prep. of trimethylenediamine in good yield by the reduction of $NH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CN$ with Na–K alloy in BuOH is described. $NH(CH_2{\cdot}CH_2{\cdot}NH_2)_2$ is obtained by the reduction of $NH(CH_2{\cdot}CH_2{\cdot}CN)_2$.

Aq. NH_3 (32%) (950 ml.) is kept below 35° while $CH_2{:}CH{\cdot}CN$ (I) (95 g.) is added dropwise, so that a second layer is not formed and temp. is < 38°. After stirring for 30 min. the H_2O is removed under vacuum. The residue is distilled, giving $NH_2{\cdot}(CH_2)_2{\cdot}CN$ (II) (30%), b.p. 77—78°/14 mm., 84—86°/18 mm., 89°/23 mm., d_4^{20} 0·9584, n_D^{20} 1·4390 (*hydrochloride*, $C_4H_8N_2,HCl$, m.p. 163—163·5°), and 2 : 2′-dicyanodiethylamine (III) (47·2%), b.p. 177—179°/14 mm., 209—211°/22 mm., d_4^{20} 1·0196, n_D^{20} 1·4630 (*hydrochloride*, $C_6H_9N_3,HCl$, m.p. 147—148°). The residue after distillation gives, upon precipitation from a mixture of H_2O, EtOH, and Et_2O, 2 : 2′ : 2″-tricyanoethylamine, m.p. 57—58° (lit., 59°). With ½ vol. of aq. NH_3, the yield of II falls to 20%. With 22% aq. NH_3, the yield of II is only 5%, whilst that of III is only 15%, and much polymer is formed. By carrying out the distillation in a current of NH_3, the yield of II is increased to 34—35%. If the distillation of pure III is carried out in a current of NH_3, and the fractional distillation in a current of N_2, the yield of II is 6—8%. By slow addition of Et_2O–HCl to III, and hydrolysis of the hydrochloride, the product is *NN-di-(2-cyanoethyl)benzamide*, $C_{13}H_{13}ON_3$, m.p. 112°. By the action of $PhSO_2Cl$ on III, *NN-di-(2-cyanoethyl)benzenesulphamide*, $C_{12}H_{13}O_2N_3S$, m.p. 88°, is obtained. III (6·2 g.) and I (8 g.) are heated in a sealed tube at 160—180° for 60 hr. Excess I is distilled off from the reaction mixture, and the residue is crystallised from a mixture of H_2O, Et_2O, and EtOH, to give 2 : 2′ : 2″-tricyanotriethylamine (40%), m.p. 57—58°. Na–K alloy (96% of technical Na with 2% of K) (30 g.) is treated with freshly-distilled II (14 g.) in dry BuOH (450 ml.), and heated at 130—140° until the metal is dissolved. After 1 hr., 150 ml. of cold H_2O is added, and the mixture is steam-distilled into a receiver containing dil. HCl. The alcohol layer is separated, and washed with dil. HCl. From the aq. extracts is obtained triethylenediamine hydrochloride (81%), m.p. 242°. The yield is lower if the II used is not freshly distilled. Using *iso*pentyl alcohol, the yield is 69%. The hydrochloride is decomposed under strongly alkaline conditions, with cooling, to trimethylenediamine (74·3%, from hydrochloride), b.p. 136—138°/756 mm., d_4^{20} 0·8634, n_D^{20} 1·4580. III is similarly reduced with Na–K alloy to give di-(3-aminopropyl)-amine (23—25%), b.p. 105—106°/5—6 mm., 114—117°/17 mm. (lit., 100°/2—3 mm.; 128—131·5°/20 mm.), d_4^{20} 0·9368, n_D^{20} 1·4848 (hydrochloride, $C_6H_{17}N_3,HCl$, m.p. 259° (decomp.) (lit., 254°), picrate, $C_6H_{17}N_3,3C_6H_3O_7N_3$, m.p. 226—227° (decomp.) (lit., 230°)).

C. A. Finch

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TA 170T24

TERENT'EV A. P.

USSR/Chemistry - Cyanides Nov 50

"Syntheses Based on the Use of Acrylic Acid Nitrile: XI. Cyanoethylation of Ethylenediamine," A. P. Terent'ev, A. N. Kost, Lab of Org Chem imeni Acad N. D. Zelinskiy, Moscow State U

"Zhur Obshch Khim" Vol XX, No 11, pp 2069-2071

Mono- and dicyanoethylated products formed as result of action of acrylic acid nitrile on diethylamine were isolated and characterized. Shows dicyanoethylated product was N, N'-di (beta-cyanoethyl)-ethylenediamine.

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eA

Sulfonation and sulfonic acids of acidophobic compounds. XII. Sulfonation of styrene and 1-phenylbutadiene. A. P. Terent'ev and A. V. Dombrovskii (Moscow State Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 1876-80(1950); cf. *C.A.* 44, 1480f, 7828c.—Sulfonation of styrene with pyridine-SO_3 occurs by intermediate formation of $PhCH:CHSO_3NHC_6H_5$; $PhCH:CHCH:CH_2$ sulfonates by a similar process in the terminal position. Heating 5.6 g. styrene, 17.6 g. pyridine-SO_3, and 6 ml. $(ClCH_2)_2$ 10 hrs. at 100° in a sealed tube, followed by treatment with NH_4OH, concn., treatment with $Ba(OH)_2$ (excess Ba removed by CO_2), and extn. with hot H_2O gave 13 g. $(PhCH:CHSO_3)_2Ba$, poorly sol. in cold H_2O; the *K* and *Na salts* are obtainable by metathesis with appropriate carbonates; the *Ag salt* appears to be too unstable to be isolated, as the ppt. obtained on addn. of $AgNO_3$ to solns. of the Na or K salts rapidly turns black. The *free acid* (from the Ba salt) is a sirup that crystallizes slowly over H_2SO_4 *in vacuo* and m. 54°, decomp. 120°. Smelting 4 g. Na salt with 8 g. dry HCO_2Na, followed by acidification, yields cinnamic acid; oxidation of the Na salt with hot $KMnO_4$ yields BzOH. Warming the Na salt with 2 parts PCl_5 gave $PhCH:CHSO_2Cl$, m. 88° (from C_6H_6), which, heated 1 hr. with satd. alc. NH_3, gave the *amide*, m. 142°, while heating in C_6H_6 with 2-aminopyridine in the presence of pyridine similarly gave the corresponding *N-2-pyridylamide*, m. 185°. Addn. of Br water to an aq. soln. of the Na salt gave upon evapn. a cryst. solid, apparently $PhCH:CBrSO_3H$ (from R(OH)). Heating 5 g. $PhCH:CHCH:CH_2$, 18.2 g. pyridine-SO_3, 6 ml. $(ClCH_2)_2$, and 0.3 g. *m*-$C_6H_4(NO_2)_2$ (as stabilizer) 4 hrs. at 100° similarly gave 49.6% $(PhCH:CHCH:CHSO_3)_2Ba$; *Na salt* (by treatment of the Ba salt with Na_2CO_3), is easily sol. in H_2O; oxidation with $KMnO_4$ yields BzOH; complete hydrogenation of the Na salt in H_2O over Raney Ni gave the satd. analog, while addn. of only 2 atoms H, followed by $KMnO_4$ oxidation, readily gave $PhCH_2CH_2CO_2H$, as well as some BzOH. G. M. K.

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CA TERENT'YEV, A.P.

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The study of pyrrolesulfonic acids by oxidative polarography. A. P. Terent'ev, L. A. Yanovskaya, and F. A. Terent'eva (Lomonosov State Univ., Moscow). *Doklady Akad. Nauk S.S.S.R.* **70**, 649-51(1950); cf. C. A. **43**, 1016, **44**, 1095a. Pyrrolesulfonic acids are not reduced at the dropping-Hg cathode, but are readily oxidized in acid solns., giving distinct polarographic curves. The oxidation potential was measured with a calomel electrode and 0.5 N KCl; the best oxidation occurred at pH 4.2-4.5 when pure Ba salts of the acids were used. *2-Pyrrolesulfonic acid* gives a half wave potential of 0.022 v.; *1-methyl-2-pyrrolesulfonic acid* gave 0.018 v., and *2,4-dimethyl-3-pyrrolesulfonic acid* gave 0.015 v.; *2,4,5-trimethyl-3-pyrrolesulfonic acid* gave 0.052 v.; *1,2,5-trimethyl-3-pyrrolesulfonic acid* gave 0.044 v. Mixts. of the 2- and the 3-sulfonic acids give sets of 2 waves corresponding to the components. The alleged *1-phenylpyrrolesulfonic acid* (cf. *Vestnik Moscov. Univ.* **1948**, No. 11, 123) gave 2 waves with half wave potentials 0.026 and 0.052 v., indicating an isomer mixt. of 2- and 3-sulfonic acids. *1-Phenylpyrrolesulfonic acid* (cf. above ref.) similarly is a mixt. of 2,3- and 2,4-disulfonic acids (0.02 and 0.042 v. half wave potentials), while *1-p-nitrophenylpyrrolesulfonic acid* (cf. above ref.) also revealed both 2- and 3-sulfo derivs. However, *1-o-toluylpyrrolesulfonic acid* was shown to be a pure 2-isomer, but *1-acetylpyrrolesulfonic acid* revealed the presence of 2- and 3-sulfo derivs. G. M. K.

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Asymmetric synthesis with the aid of catalysts deposited on right and left quartz. A. P. Terent'ev, E. I. Klabunovskii, and V. V. Patrikeev (Moscow State Univ.). *Doklady Akad. Nauk S.S.S.R.* **74**, 947-50(1950). -The following reactions, characterized by the observed rotation of a 20-cm. layer of the product, were carried out on metals deposited by either reduction or evapn. on either left (*l*) or right (*r*) quartz powder. (1) *Sepn. of racemates.* Dehydration and dehydrogenation of *dl*-MeCH(OH)Et vapor was carried out in a flow system, with dry N_2 as carrier gas, on (*l*), with Cu (reduced at 300°), at 360°, rotation (due to unreacted MeCH(OH)Et) −0.112°; with Ag (sputtered), 350°, −0.080°; with Ni (reduced at 300°), 365° −0.120°; with Pd (sputtered) 280°, 0.080°; on (*r*), with Cu (reduced at 300°), 360°, 0.160; Cu (sputtered) 460°, 0.180°; Ag (sputtered) 470°, 0.100°; Pd (from $PdCl_2$) 360°, 0.060°; Ni (reduced at 300°) 388°, 0.110°. Isomerization of *dl*-MeCH.CH_2.O vapor to Me CO and EtCHO in a flow system, with N_2 as carrier gas, on (*l*), with Cu (sputtered) at 180°, rotation (due to unreacted substance) −0.055, Ag (sputtered) 150°, −0.052°; Pd (sputtered) 186°, −0.031°, on (*r*), with Cu (sputtered) 230°, 0.031°. (2) *Destruction of racemate with formation of a new center of asymmetry.* Dismutation of 2-methylcyclohexanone (I), according to 3 I + 2 2-methylcyclohexanol + *o*-MeC_6H_4OH, in a flow system in N_2, on (*l*), with Pt (sputtered) 214°, rotation (of the catalyzate fraction b. 158-70°) −0.042°; Pd (sputtered) 320°, −0.025°; on (*r*), with Pt (from H_2PtCl_6) 320°, 0.028°, Pd (sputtered) 305°, 0.030°. (3) *Absolute asymmetric synthesis.* Hydrogenation of α-pinene in decahydronaphthalene soln., on (*l*), with Ni (reduced at 350°) 135°, rotation (of the fully hydrogenated product) 0.046°; on (*r*), with Ni, 135°, −0.028°. Hydrogenation of PhCH:CPhCO$_2$Et to PhCH$_2$CHPhCO$_2$Et in decahydronaphthalene soln., on (*l*), with Ni (reduced at 350°) 135°, rotation (of the fully hydrogenated product) 0.041°; on (*r*), with Ni, 135°, −0.084°. The latter are instances of syntheses of optically active compds. from compds. without an asym. C atom.

N. Thon

1957

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