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MICHALSKI, J.

Reactions of alkyl alkanethiosulfonates with trialkyl phosphites. A new synthesis of *O,O,S*-trialkyl thiophosphates and esters of alkanesulfinic acids. Jan Michalski, Jan Wieczorkowski, and Tomasz Modro (Politech., Łódź, Poland). *Roczniki Chem.* 32, 1409-11(1958)(in English). —Et butanethiosulfonate was treated with tri-Bu phosphite (1:1) at 20-5°. The products were distd. *in vacuo* to yield 89% *O,O*-di-Bu *S*-ethylthiophosphate (I), 87-9°, n_D^{20} 1.4524, and 90% Bu butanesulfonate (II), $b_{0.2}$ 52-4°, n_D^{20} 1.4444. The structure of I was confirmed by reaction with Cl in aq. soln. to give ethanesulfonyl chloride, which was transformed into ethanesulfonyl *p*-toluidide, m. 79-80°. III was hydrolyzed in 1.4*N* alc. aq. 1.4*N* to Na butanesulfinate. The later was condensed with 2,4-dinitrochlorobenzene to Bu 2,4-dinitrophenyl sulfone, m. 91-2°. The reaction between I and tri-Et phosphite was carried out in an analogous way. Fractional distn. of the products gave 3 fractions: an azeotropic mixt., 83% Et butanesulfinate (IV), and 17% *O,O,S*-tri-Et thiophosphate (V), b_{11} 80-83°, n_D^{20} 1.4385, and b_{12} 115°, n_D^{20} 1.4572. The last fraction was crystd. to give Bu Et sulfone (VI), m. 49-50°. Taking into account the azeotrope, the over-all yields of IV, V and VI were 67, 89 and 9%, resp. The reaction schemes are given.

A. Kręglewski

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MICHALSKI, J.

Organophosphorus compounds of sulfur and selenium. IX. Addition of dialkoxyphosphinylsulfenyl chlorides and alkylalkoxyphosphinylsulfenyl chlorides to symmetrical olefins. Barbara Lenard-Borecka, Jan Michalski, and Stanisław Musierowicz (Polish Acad. Sci., Łódź). *Rocznik Chem.* 32, 1301-9(1958)(English Summary); cf. *C.A.* 51, 2535h; 53, 10013h, 15956e.—Dialkoxyphosphinylsulfenyl chlorides, $(RO)_2P(O)SCl$ (I), and alkylalkoxyphosphinylsulfenyl chlorides, $R(RO)P(O)SCl$ (II), add spontaneously and exothermally to C_2H_4 (III), $(Me_2C:)_2$, and cyclohexene. The reaction with stilbene requires irradiation by sunlight. E.g. 20.4 g. $(EtO)_2P(O)SCl$ in 30 ml. C_6H_6 with III (gas) at 5-10° gave, after distn., 19.7 g. (84.5%) $(RO)_2P(O)SCH_2CH_2Cl$ (R = Et) (IV), $b_{0.05}$ 68°, n_D^{20} 1.4785, d_{20} 1.2276. The following analogs of IV were prepd. (R, b.p./mm., n_D^{20}, d_{20}, % yield): Pr, 72°/0.005, 1.4751, 1.1657, 77; *iso-Pr*, 100°/0.9, 1.4680, 1.1462, 86; *Bu*, 95.2°/0.01, 1.4731, 1.1208, 66. The following were also prepd.: $Et(EtO)P(O)SCH_2CH_2Cl$, 94°/0.05, 1.4958, 1.1041, 74; $(EtO)_2P(O)SCMe_2CMe_2Cl$, 87/0.02, 1.4840, 1.1106, 60; $(BuO)_2P(O)SCH_2CH_2Cl$, 80°/0.05, n_D^{20} 1.4885, d_{20} 1.1333, 75; $(PrO)_2P(O)SCMe_2CMe_2Cl$, 80°/0.01, n_D^{20} 1.4858, d_{20} 1.0908, 40; $(EtO)_2P(O)SC_6H_{10}Cl$-*o*, 91°/0.1, 1.4963, d_{20} 1.1948, 62; $(EtO)_2P(O)SCHPhCl$, m. 98-9°, yield 58%. The structure of IV was verified by synthesis by Morrison's method (*C.A.* 50, 789h), and by oxidative chlorination according to Stirling, (*C.A.* 52, 14556g). The compds. inhibit cholinesterase activity and do not possess any blistering property.

Czesław Bankiewicz

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Distr: 4E2c(j)/4E3d

Organophosphorus compounds of sulfur and selenium. Addition of dialkoxphosphinylsulfenyl chlorides and alkylalkoxyphosphinylsulfenyl chlorides to symmetrical olefins. J. Michalski, B. Borecka, and S. Musierowicz (Politech. Łódź, Pol.). *Bull. acad. polon. sci., Sér. sci. Chim., géol., et géograph.* 6, 159–63(1958)(in English).—The addn. of organophosphorus compds. with a P(O)SCl group (cf. *C.A.* 52, 9945*h*) to sym. olefins has been studied. By addn. of $(RO)_2P(O)SCl$ (I) or $R'(RO)P(O)SCl$ (II) to C_2H_4 (III), $Me_2C{:}CMe_2$ (IV), or cyclohexene, the following compds. have been prepd. (yield, b.p./mm., n_D^{20}, d_{20} given): $(EtO)_2P(O)SCH_2CH_2Cl$ (V), 84, 68°/0.06, 1.4785, 1.2276 (Morrison, *C.A.* 50, 780*h*); $(EtO)_2P(O)SCMe_2CMe_2Cl$, 60, 67°/0.02, 1.4840, 1.1106; $(PrO)_2P(O)SCH_2CH_2Cl$, 77, 72°/0.005, 1.4751, 1.1057; $(PrO)_2P(O)SCMe_2CMe_2Cl$, 40, 86°/0.01, 1.4858, 1.0908 at 25°; $(iso\text{-}PrO)_2P(O)SCH_2CH_2Cl$, 70, 100°/0.9, 1.4680, 1.4262; $(BuO)_2P(O)SCH_2CH_2Cl$, 66, 92.5°/0.015, 1.4731, 1.1208; $Et(EtO)P(O)SCH_2CH_2Cl$ (VI), 74, 640/0.05, 1.4958, 1.1941; $Et(BuO)P(O)SCH_2CH_2Cl$, 75, 80°/0.05, 1.4885, 1.1333 at 25°; $(EtO)_2P(O)SR$ (R = 2-chlorocyclohexyl), 62, 91°/0.1, 1.4963, 1.1948; $PhCHClCHPhSP(O)(EtO)_2$ (VII), 58, m. 98–9° (repeatedly from C_6H_6), —, —. E.g.: I (R = Et) (20.4 g.) dild. with 30 ml. C_6H_6, kept at 5–10°, pure III introduced until the yellow color disappeared, the soln. washed with H_2O, satd. $NaHCO_3$, and H_2O, dried, and the C_6H_6 evapd., gave V. The synthesis of VI was performed at 10–20°; that of VII required 3 days' exposure to light.

J. Stecki

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POLAND / Organic Chemistry. Synthetic Organic Chemistry.

G-2

Abs Jour: Ref Zhur-Khimiya, No 23, 1958, 77747.

Abstract: 2-$C_5H_4NCH(CH_3)PO(OC_2H_5)_2$ (II), mp 141-143°, which differs from the isomeric picrate of I (R = $P(O)(OC_2H_5)_2$); II has a bp of 95-97°/0.05mm, $n^{25}D$ 1.4920. I (R = $P(O)(OH)_2$) and I (R = 2-C_5H_4-$NCH(CH_3)P(O)(OH)_2$) form salts with $C_6H_5CH_2NH_2$ having mp's of 179-180 and 171°, respectively. -- V. Gilyarov.

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

Abs Jour: Ref Zhur-Khimiya, No 23, 1958, 77747.

Abstract: $n^{25}D$, d_4^{25}, and the mp in °C of the picrolonate are given in that order): $P(O)(OC_2H_5)_2$, 40 (60 with C_2H_5ONa), 101/0.05, 1.4938, 1.1156, 91-92; $P(O)(OC_3H_7)_2$, 99/0.05, 1.4812, 1.0591, 134-135 (decomp); $P(O)(OC_4H_9\text{-}n)_2$, 43 (65 with C_2H_5ONa), 132-0.2, 1.4841, 1.0460, --; $P(O)(OC_2H_5)C_6H_5$, 59, 143-145/0.1, 1.5560, 1.1419, picrate mp 158-159°; $P(O)(CH_2C_6H_5)_2$, 92, --, mp 118°, -, -, -, picrate mp 158-160°; $P(S)(OC_2H_5)_2$, 58, 97/0.05, 1.5101, 1.1085 (at 30°), -. The structure of I is proved by forming the picrate

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G-2

POLAND / Organic Chemistry. Synthetic Organic Chemistry.

Abs Jour: Ref Zhur-Khimiya, No 23, 1958,77747.

Author : Maruszewska-Wierzorkowska, E. and Michalski, J.
Inst : Polish Academy of Sciences.
Title : Synthesis of Organophosphorus Compounds Containing Pyridylalkyl Radicals by the Addition of Dialkyl Phosphites and Their Structural Analogs to 2-Vinylpyridine.

Orig Pub: Bull Acad Polon Sci, Ser Sci Chim Geol et Geograph, 6, No 1, 19-21 (1958) (in English).

Abstract: The addition of R'R''P(X)H (R', R'' = alkyl or alkoxyl, X = O or S) to 2-vinylpyridine in the presence of C_2H_5ONa at 100° or in the absence of C_2H_5ONa under milder conditions gives 2-$C_5H_4NCH_2R$ (I) (R, the yield in %, bp in °C/mm,

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vacuo, and the residue distd. gave 96% XVI. XVI (3.7 g.) in 10 ml. C_6H_6 treated dropwise with 2.1 g. SO_2Cl_2 in 10 ml C_6H_6 at −2 to 0°, the filtered soln. evapd., and the residue distd. gave 48% XV, b_{13} 104°, $n^{20}D$ 1.4668. Alc. NaOEt (3.35 g. Na in 100 ml. alc.) treated with 33 g. $(PhCH_2)_2PHO$ in 200 ml. alc., the mixt. stirred with portionwise addn. of 5.2 g. S at 30–5°, the stirring continued 2 hrs., the filtered soln. evapd. *in vacuo*, and the product crystd. (alc.) gave $(PhCH_2)_2PSONa$, m. 232–6°. The salt (31 g.) in 200 ml. H_2O decompd. by excess HCl at 0° gave *$(PhCH_2)_2PS$-OH* (XVII), m. 190–1° (1:10 alc.-C_6H_6). XVII (5.24 g.) in 120 ml. CCl_4 at −25 to −20° treated dropwise with 2.7 g. SO_2Cl_2 in 30 ml. C_6H_6, the solvent evapd. at 0° *in vacuo* with pptn. of S, the filtered soln. hydrolyzed by addn. of a few drops of water, and the product crystd. (C_6H_6) gave authentic $(PhCH_2)_2PO_2H$, m. 191–3°. C. R. Addinall

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✓ Organophosphorus compounds of sulfur and selenium. X. Action of sulfuryl chloride on alkyl hydrogen alkylphosphonothioates and dialkylphosphinothioic acids. Synthesis of *p*-alkoxy-*p*-alkyloxophosphoranesulfonyl chlorides. Cz. Borecki, J. Michalski, and St. Musierowicz (Tech. Univ., Łódź, Poland). *J. Chem. Soc.* 1958, 4081–5; cf. preceding abstr.—SO_2Cl_2 with R(R'O)PSOH (I) gave RPO(OR')SCl (II) or [RPO(OR')S]$_2$ (III). R_2PSOH (IV) with SO_2Cl_2 gave R_2POCl (V) or $R_2PSOPOR_2$ (VI). The nomenclature used is based on the hypothetical phosphorane PH_5 and the radical phosphenyl, $PH_2(O)^-$. SO_2Cl_2 (27 g.) in 30 ml. C_6H_6 added dropwise to 30.8 g. I (R = R' = Et) (VII) in 100 ml. C_6H_6 at −5 to 0° (external cooling), the solvents and gaseous products evapd. *in vacuo*, and the products distd. gave 70% II (*R* = *R'* = *Et*) (VIII), $b_{0.5}$ 33–4°, n_D^{20} 1.4800, d_{20} 1.2312. $BuEtPO_2H$ (74.3 g.) in 150 ml. BuOH contg. 11 g. Na treated slowly with 15 g. S with cooling, the filtered soln. evapd. *in vacuo*, the crude Na salt taken up in 50 ml. H_2O and acidified with 90 ml. 20% HCl, the free acid extd. 3 times with 70 ml. C_6H_6, the exts. evapd. *in vacuo*, and the residue distd. gave 51.5 g. *Bu(EtO)PSOH* (IX), $b_{0.05}$ 76°, n_D^{20} 1.4821, d_{20} 1.0683. IX (18 g.) in 60 ml. C_6H_6 treated dropwise at −10 to −5° (external cooling) with 13.5 g. SO_2Cl_2 in 40 ml. C_6H_6, the solvent evapd., and the residue distd. *in vacuo* yielded 55% II (*R* = *Et*, *R'* = *Bu*), $b_{0.4}$ 74°, n_D^{20} 1.4528. VIII (26.4 g.) heated at 60–70°/1 mm. with pptn. of S and distn. gave 15.5 g. material, redistd. *in vacuo* to give the known EtPO(OEt)Cl, $b_{0.5}$ 35–6°, n_D^{20} 1.4402. SO_2Cl_2 (5.15 g.) in 20 ml. C_6H_6 added dropwise to 11.75 g. VII in 60 ml. C_6H_6 at 0°, the solvent and gaseous products removed *in vacuo*, and the residue distd. gave 98% III (*R* = *R'* = *Et*) (X), n_D^{20} 1.5061. VIII (9.47 g.) in 100 ml. C_6H_6 added dropwise to 11.75 g. VII in 60 ml. C_6H_6 at 0°, the solvent and gaseous products removed *in vacuo*, and the residue distd. gave 98% III (*R* = *R'* = *Et*) (X), n_D^{20} 1.5061. VIII (9.47 g.) in 100 ml. C_6H_6 added dropwise to 7.79 g. VII in 70 ml. C_6H_6 at −5 to 0°, the solvent evapd. *in vacuo*, and the residue distd. yielded 98% X. X (15.2 g.) heated at 120°/0.05 mm. and the product distd. yielded 75% authentic EtPS(OEt)OPEtO(OEt) (XI). The structure of the anhydride XI was confirmed by comparison with compds. prepd. by the action of H_2S on alkyl alkylphosphonochloridates, RPO(OR')Cl (XII), in the presence of tertiary bases according to Michalski (*C.A.* 50, 10641*h*). S (16.2 g.) added portionwise to 63.5 g. Et_2PCl in 250 ml. C_6H_6 at 18–24°, the solvent evapd., and the residue distd. yielded 89% *Et_2PSCl* (XIII), b_9 94–5°, n_D^{20} 1.5292. XIII (45 g.) treated dropwise with 25 g. NaOH in 50 ml. H_2O at 30–40°, the soln. acidified with 20% HCl and the product added to the 3 washings of the aq. layer with 50 ml. C_6H_6, the soln. evapd., and the residue distd. yielded 78% *Et_2PSOH* (XIV), $b_{0.12}$ 67–8°, n_D^{20} 1.5257; *cyclohexylamine salt* m. 145–7°. SO_2Cl_2 (14.6 g.) in 50 ml. C_6H_6 added dropwise to 15 g. XIV in 50 ml. at −6 to −2° with pptn. of S, the solvent evapd., and the residue distd. gave 10.2 g. V (*R* = *Et*) (XV), b_{10} 90–8°, n_D^{20} 1.4682. SO_2Cl_2 (6.5 g.) in 20 ml. C_6H_6 added dropwise to 13.4 g. XIV in 30 ml. C_6H_6 at 0°, the mixt. kept 1 hr. at room temp., the filtered soln. evapd., and the residue distd. yielded 59% VI (*R* = *Et*) (XVI), $b_{0.07}$ 94–5°, n_D^{20} 1.5030. XIV (8 g.) in 8 ml. C_6H_6 added dropwise to 8.1 g. XV in 7 ml. C_6H_6 at 15–20°, the HCl and solvent evapd. *in*

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Reactions of thiono esters of phosphorus with halogens and sulfuryl chloride. J. Michalski and Alexandra Skowronska (Inst. Technol., Lodz, Poland). *Chem. & Ind.* (London) 1958, 1199–1200; cf. *C.A.* 52, 9945*h*.—Esters of thio acids of P contg. the >P(S)OR (I) group (R = alkyl) react readily with Cl, Br, or SO_2Cl_2 to give phosphinylsulfenyl halides, >P(O)SX, where X = Cl. Formation of a mesomeric complex probably is involved as an intermediate. These reactions are useful in prepg. the I group and also as a test to distinguish between thiono esters (I) and thiolo esters, >P(O)SR, which are known to react with Cl to give RSCl and >P(O)Cl (Stirling, *C.A.* 52, 14556*g*). Addn. of 1 molar equiv. Cl in CCl_4 to $(EtO)_2P(S)OEt$ (II), with the temp. kept at −5°, gave 50% $(EtO)_2P(O)SCl$, $b_{0.3}$ 49–50°, n_D^{20} 1.4672. Similarly was obtained 70% $(BuO)_2P(O)SCl$, $b_{0.07}$ 73–4°, n_D^{20} 1.4665 (SO_2Cl_2 in C_6H_6 at 0°). II and Br in C_6H_6 gave $(EtO)_2P(O)SBr$, unstable, which with $CH_2:CH_2$ gave $(EtO)_2P(O)SCH_2CH_2Br$, $b_{0.3}$ 86°, n_D^{20} 1.4900. PhOP(S)$(OEt)_2$ with SO_2Cl_2 gave 50% EtO(PhO)P(O)SCl $b_{0.3}$ 97–8°, n_D^{20} 1.5335, which with $CH_2:CH_2$ gave 75% $P(O)(OEt)(OPh)SCH_2CH_2Cl$, $b_{0.3}$ 124–5°, n_D^{20} 1.5340.

Rip G. Rice

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

Abs Jour: Ref Zhur-Khim., No 24, 1958, 81722.

84, 125-126; $[(CH_3)_2N]_2P(S)NHCSNHC_6H_4$, 78, 130.
Communication V, see R. Zh Khim., 1958, 64543.

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Abs Jour: Ref Zhur-Khim , no 24, 1958, 81722

a large R, are not stable, and decompose on $(R'_2NPO_2)_n$ and RSCN. By the action of RONa on R'_2NPOCl_2, or by the reaction of R'_2NH with $ROPOCl_2$ the following I were synthesized (here and further for describing the products are given R, yield in %, b.p. in °C./mm $n^{20}D$, D^{20}_4): I-a, C_3H_7, 72, -, 1 4381, 1 1432; I-a, C_4H_9, 69, 82-84/1, 1 4408, 1 0926; I-b, C_4H_9, -, 96/1 3, 1.4436 (26°), -. In the same way was obtained II-a, C_2H_5, 66, 46.5-47.5/0.4, 1.4947, -,. To the solution of 0.14 moles of KCNS in 200 ml of CH_3CN, 0.14 moles of I-a is added, after one hour CH_3CN is distilled under vacuum, 100 ml of benzene is added, from the filtrate III-a was separated, C_2H_5, 35, 59-61/0.12, 1.4930, 1.1529. In the same

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POLAND/Organic Chemistry Synthetic Organic Chemistry. G-2

Abs Jour: Ref Zhur-Khim., No 24, 1958, 81722.

Author : Michalski J., Strzelecka H , Wieczorkowski J.
Inst :
Title : The Phosphororganic Compounds Containing Sulfur and Selenium. VI Amido Esters and Amides of Isothiocyanophosphoric and Isothiocyanothiophosphoric Acids.

Orig Pub: Roczn. chem., 1957, 31, No 3, 879-891

Abstract: By the reaction of KCNS on $(RO)(R'_2N)P(O)Cl$ (I) (here and further aR' = CH_3, bR' = C_2O - /sic/), $(RO)(R'_2N)P(S)Cl$ II, $(R'_2N)_2P(O)Cl$ and $(R'_2N)_2P(S)Cl$, were obtained the corresponding products by substituting chlorine for the NCS group (III-VI). The structure of III-VI was verified by the synthesis of addition compounds of amines. III, especially with

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MICHALSKI, J.

POLAND / Organic Chemistry. Synthetic Organic Chemistry. G

Abs Jour: Ref Zhur-Khimiya, No 18, 1958, 61033.

Author : Jan Michalski, Czeslaw Krawiecki.
Inst : -
Title : Dialkylselenophosphites of $(RO)_2PSeH$.

Orig Pub: Roczn. chem., 1957, 31, No 2, 715-716.

Abstract: $(C_2H_5O)_2PSeH$ (I), boiling point 42 to 43°/0.6 to 0.7 mm, $n^{25}D$ = 1.4965, d_4^{25} = 1.3650, was prepared by introducing dry H_2Se into cooled solution of $(C_2H_5O)_2PCl$ and equivalent amount of tertiary amine in C_6H_6. I reacts with SO_2Cl_2 producing $(C_2H_5O)_2P(Se)Cl$, boiling point 62°/1 mm, $n^{25}D$ = = 1.4974, d_4^{25} = 1.4411. I adducts CH_2=CHCN with

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G

Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64543.

2 g. cyclohexamine in 10 ml. ligroin, yielding (VII) (R=iso-C_3H_7, R'=cyclohexyl) 92%, m.p.106° (in cyclohexane). To a solution of 2.11 g. (IVb) (R=C_2H_5) in 4 ml. ligroin, there is added 1 g benzylamine in 5 ml ligroin, yielding (VIII) (R=C_2H_5; R'=$C_6H_5CH_2$) 88%, m.p. 78° (in ligroin or hexane). To a solution of 3.16 g. (IVb) (R=C_2H_5) in 40 ml n-hexane, there is added 1.5 g. cyclohexylamine in 10 ml. n. hexane, yielding (VIII) 86%, m.p. 55° (in ligroin). For previous report, see RZhKhim, 1957, 73718.

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G

Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64543.

there is gradually added a solution of 7 g. dimethylamine in 150 ml C_6H_6, during 24 hours. There is liberated 4.5 g of (VI), m.p. 112°; from the filtrate can be drawn (V) (R=C_2H_5), 67, 98/21, 1.4191,-. To a solution of 17.2 g. (IX) in 50 ml C_6H_6, there is added gradually 10 g. dimethylamine in 200 ml C_6H_6 at 10-12°, yield of (V) (R=C_2H_5) 78%. To a solution of 3.9 g. (IVa) (R=C_2H_5) in 40 ml ligroin are added 2.15 g benzylamine in ligroin. The remainder, after driving off the ligroin, is dissolved in C_6H_6 and washed with 15 ml 4% HCl, 5% $NaHCO_3$, and water, yielded (VII) (R=C_2H_5), R' = $C_6H_5CH_2$) 81%, m.p. 89° (in cyclohexane). Similarly derived is (VII) (R = C_2H_5) R'= cyclohexyl), yield 75%, m.p. 85° (in cyclohexane). To a solution of 4.47 g. (IVa) (R=iso-C_3H_7) in 30 ml ligroin, there is added a solution of

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G

Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64543.

of 0.4 moles of (I) in 500 ml. C_6H_6, the gradual addition during 50 hours at 0° of a solution of 46.2 g of (III) (R = C_2H_5) in 100 ml. C_6H_6, followed by centrifuging and distilling the product, gives (IVb) (R = C_2H_5), 73, 73-74/0.6, 1.5220, 1.1906. Similarly, with (IVb) (R = C_4H_9), 72, 109/0.7, 1.5088, 1.0981. To a solution of 9.7 g. of KCNS in 150 ml. of acetone, is added 17.3 g. (C_2H_5O) P(O)Cl (IX), and from the filtrate is extracted (IVa) (R=C_2H_5), yield 56%; in the presence of acetone, yield 66%. By the same method, yields are: (IVa) (R=C_4H_9), 57%, (IVb) (R=C_2H_5) 60%, (IVb) (R=C_4H_9) 62%. From 32.5 g. $(C_3H_7O)_2P(S)Cl$ and a solution of 14.6 g. KCSN in 200 ml of acetonitrile, there is produced (IVb) (R=C_3H_7), 68. 99-102/1.8, 1.5120, 1.1275. To a solution of 14.7 g. (IVa) (R=C_2H_5) in 50 ml C_6H_6,

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G

Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64543.

upon the reaction of (IVa) with dimethylamine, there are produced $(RO)_2P(O)N(CH_3)_2$ (V) and $(CH_3)_2NH.HSCN$ (VI). Upon the reaction of (IVa) with primary amines, there is produced the thiourea $(RO)_2P(O)NHC(S)NHR'$ (VII). (VIb) hydrolizes with more difficulty. Upon its reaction with amines, there is produced $(RO)_2P(S)NHC(S)NHR'$ (VIII). To the solution of 27.6 g (II) ($R=C_2H_5$) in 100 ml. C_6H_6, there is gradually added at 50° a solution of 0.25 moles of (I) in C_6H_6. Stir for an hour, then after 24 hours there can be separated (IVa) ($R=C_2H_5$) (here and later in the report, yields are given in %, and b.p. in °C/mm, $N^{20}D$, d_4^{20}): 80, 64-65/0/6, 1.4791, 1.1870. Similarly derived is (IVb) where $R = C_3H_7$, 74, 72-73/0.2, 1.4751, 1,1213; (IVa) ($R = iso-C_3H_7$), 77, 63-64/0.2, 1.4690, 1.1076; (IVa) ($R = C_4H_9$) 88, 86-87/0.12, 1.4718, 1.0757. To a solution

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POLAND/Organic Chemistry. Synthetic Organic Chemistry. G

Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64543.

Author : Michalski, Jan; Wieczorkowski, Jan
Inst :
Title : Phosphoroorganic Derivatives of Sulfur and Selenium. V. The Action of Thiocyanogen on Dialkylphosphites and Thiophosphites. Dialkyl Isothiocyanophosphates and Isothiocyanophosphates.

Orig Pub: Roczn. chem. 1957, 31, No 2, 585-600.

Abstract: The action of thiocyanogen (I) on $(RO)_2POH$ (II) or $(RO)_2PSH$ (III) produces HSCN and, with a yield of 70-80%, $(RO)_2P(Z)NCS$ (IV), where Z = O (IVa), or S (IVb). (IVa) and (IVb) can also be derived by the action of KCNS on $(RO)_2P(O)Cl$ or $(RO)_2P(S)Cl$. Upon hydrolysis of (IVa), HSCN is produced, as well as $(RO)_2P(O)OH$;

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G

POLAND/Organic Chemistry. Synthetic Organic Chemistry.

Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64546.

Author : Michalski Jan, Skowronska Aleksandra
Inst :
Title : Dialkyl Ethylpyrophosphonates C H (RO)P(O)-O-P(O)(RO)C H

Orig Pub: Roczn. chem. 1957, 31, No 1, 301-303

Abstract: Describes the means of getting $C_2H_5(RO)P(O)(OR)$-R = C_2H_5 (I), iso-C_3H_7 (II), C_4H_9 (III), by way of hydrolizing the corresponding C_2H_5P (O)(OR)Cl (IV) in the presence of pyridine (V) (see RZhKhim, 1958, 8038). To 0.2 moles of (IV) (R=C_2H_5), is added drop by drop and with stirring 0.106 moles of water and 0.206 moles of (V), at 0-2°, after 20 hours (~ 20°); the precipitate is washed in C_6H_6 and (I) is separated

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Abs Jour: Ref Zhur-Khim., No 13, 1958, 43439.

1.4486 (20); II and V, $(C_4H_9O)_3PS$, 42, 104/0 6, 156-157/11, 1.4497 (20); I and VI, IX, 96, and $(CH_3CO)_2S$, 97, 50/12, 61-62/20, 1.4820 (21); III and VII, $(C_2H_5O)_3PS$, 89, 148-150/0.1, — , MP 48°, and $(C_2H_5O)_4P_2O_2S$ (X), 77, 82-84/0.04, 1.4502 (25); I and VII, X, 75, $(C_2H_5O)_2P(O)SC_2H_5$ (XI), 31, 112/11, 1.4570 (20), and IX, 37; I and VIII, $C_4H_9SP(O)(OC_2H_5)_2$, 95, 131-133/11, 1.4580 (20), XI, 41; and IX, 36; IV and VII, $C_4H_9(C_2H_5O)_3P_2O_2S$, 81, 117-118/0.05, 112/0.03, 1.5065 (20), d_4^{20} 1.2050; XI, 34, IX, 37. The assumption is made of an intermediate formation of the unstable [$RO(R')P^+(SX)(OR)S$-Y$^-$], wherein R = alkyl or aryl, R' = alkoxyl, aryl or phenoxyl, X = CN, CH_3CO, $(C_2H_5O)_2P(O)$, C_4H_9, Y = CN, CH_3CO, $(C_2H_5O)_2P(O)$. The complex decomposes

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G-2

POLAND/Organic Chemistry. Synthetic Organic Chemistry.

Abs Jour: Ref Zhur-Khim., No 13, 1958, 43439.

Author : Michalski J., Wieczorkowski J.
Inst : ~~Polish Academy~~ of Sciences.
Title : The Action of Free Thiocyanogen, Diacyl Disulfides, Bis-(Dialkoxyphosphinyl) Disulfides on Esters of Acids of Trivalent Phosphorus.

Orig Pub: Bull. Acad. polon. sci., 1957, cl. 3, 5, No 9, 917-921.

Abstract: Investigation of the reactions of $(C_2H_5O)_3P$ (I), $(C_4H_9O)_3P$ (II), $(C_6H_5O)_3P$ (III) and $C_6H_5P(OC_2H_5)_2$ (IV) with $(SCN)_2$ (V), $(CH_3COS)_2$ (VI), $[(C_2H_5O)_2P(O)S]_2$ (VII) and $C_4H_9SSP(O)(OC_2H_5)_2$ (VIII). Listing initial substances, reaction products, yield in %, BP in °C/mm (in parentheses temperature in °C): I and V, $(C_2H_5O)_3PS$ (IX), 62, 93.5/13.

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MICHALSKI, I.

27 7 5

Action of hydrogen sulfide on dialkyl chlorophosphites. I. Michalski and Cz. Kamieński (Inst. Tech. Politech., Łódź, Poland). Chem. & Ind. (London) 1957, 1822-4, cf. C.A. 50, 10641A.—In the presence of pyridine, $(EtO)_2PCl$ (I) with 0.5 mole H_2S gives $(EtO)_2PHS$ (II) without formation of $[(EtO)_2P]_2S$ (III), but in the presence of a stronger base such as Et_3N, I gives III as well as II. I with II in the presence of Et_3N gives III in good yield, but no III can be isolated from the mixt. in the presence of excess H_2S, since III reacts with H_2S to give II. III, $b_{0.01}$ 66-8°, reacts vigorously with H_2O to give $(EtO)_2POH$ and $(EtO)_2PSH$, is oxidized in air to $[(EtO)_2P(O)]_2S$, b_1 86-8°, and leads to amide formation when added to a primary amine and carboxylic acid mixt.

R. H. Loeppert

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MICHALSKI, J.

MARUSZEWSKA-WIECZORKOWSKA, E.; MICHALSKI, J.; SKOWRONSKA, A.

"Organophosphorous compounds with an active methylene group. II. Dialkyl 2-pyridylmethylphosphonates $(RO)_2P(O)CH_2(2\text{-}C_5H_4N)$ and dialkyl 2-pyridylmethylthiophosphonates $(RO)_2P(S)CH_2(2\text{-}C_5H_4N)$."

p. 1197 (Roczniki Chemii) Vol. 30, no. 4, 1956
Warsaw, Poland

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

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MICHALSKI, JAN

Jan Michalski and Aleksandra Skowronska: "Organophosphorous [illegible] and Selenium. III. Dialkyethylthiopyrophosphonates [illegible] Action of Hydrogen Sulphide on Alkyl Dihydrophosphonochloridates [illegible]." Rocznik Chemii, Vol 30, No 3, Warsaw, 19[illegible]. Published [illegible] Chemistry, Lodz Polytechnic.

*Parts of this article were given at the XIV International Congress of Pure and Applied Chemistry in Zurich, July 19[illegible].

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POLAND/Optics - Spectroscopy

K-7

Abs Jour : Ref Zhur - Fizika, No 2, 1959, No 4464

Author : Michalski J., Mierzecki R., Rurarz E.
Inst : -
Title : Raman Spectra of Tetraethyl Thiopyrophosphate, Triethyl Phosphate and Triethyl Thiophosphate

Orig Pub : Roczn. chem., 1956, 30, No 2, 651-653

Abstract : Raman spectra were plotted of tetraethyl thiopyrophosphate (I), triethyl phosphate (II), triethyl thionophosphate (III) and triethyl thiophosphate (IV) and the resultant spectra were compared. The Raman spectra of I exhibit substantial differences from the spectrum of IV, but in many features it is similar to the spectra of II and III. These facts suggest that the most probable structure of the tetra alkyl thiopyrophosphates (prepared by the action H_2S on dialkyl phosphor chlorides (V) in the presence of tersiary base or condensation of V with salts of dialkyl thio phosphoric acid) can be expressed by the following formula:

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Jan Michalski and Barbara Lenarc: "Thiophosphorylation of [illegible] Condensation and Addition of Dialkyl Phosphinylsulphonyl Chloride [illegible]," Roczniki Chemii, Vol 30, No 2, Warsaw, 1956. Published from the Chair of Organic Chemistry, Lodz Polytechnic, and from the Research Laboratory of Organic Synthesis, Polish Academy of Sciences, 27 [illegible].

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[illegible] Rocznik Chem. 29, 1141-4 (1955); cf. C.A. 3, 893; 45, 1997d.—2,6-Bis(2-hydroxyethyl)pyridine (I), b_{15} 185-6° and 2-methyl-6-(2-hydroxyethyl)pyridine (II), b_{16} 126-7°, were prepd. by condensation of 2,6-lutidine with CH_2O. I and II gave, resp., by dehydration in presence of KOH 2,6-divinylpyridine (III), b_{16} 88-9°, n_D^{25} 1.5710 [picrate, m. 140.5° (from dil. alc.)], and 2-methyl-6-vinylpyridine (IV), b_{11} 73°, n_D^{25} 1.5320 [picrate, m. 160.5° (from dil. alc.)]. The following 2,6-bis(2-substituted-ethyl)-pyridines were prepd. from III (substituent, b.p./mm. and m.p. picrate given): p-$MeC_6H_4SO_2$, — (m. 55), —; PhS, 149-51°/760, 87-8°; EtO, 116°/0.5, 154-5°; Et_2N, 125-8°/18, 62-3°; morpholino, 116°/0.5, 136-7°. From IV were prepd. similar compds.: $PhSO_2$, — (m. 136°), 172-3°; BuS, 197-8°/0.5, 83-4°; morpholino, — (m. 126-7°), 164-5° (dipicrate). A. Sementsov

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6

Action of hydrogen sulfide on chlorophydrides of organic acids of tri- and quinquevalent phosphorus. Synthesis of new types of anhydrides of organic thio acids of phosphorus. New method of synthesis of dialkyl thiophosphites and their analogs. Jan Michalski (Inst. Technol., Łódź, Poland). *Roczniki* [illegible] 980-3(1956)(English summary).—Org. deriva. of the structure ABP(O)Cl (I) with H_2S in the presence of a tertiary amine give *pyro deriva.* of the type $[ABP(O)]_2S$. I (A = B = Me_2N) gave 16% of a compd., $b_{0.4}$ 91°, n_D^{20} 1.4675, d_{20} 1.1443. I (A = *Et*, and B = *EtO*, *PrO*, *BuO*, *Et*, and *Bu*) yielded the following products, resp. (% yield, b.p./mm., n_D^{20}, and d_{20} given): 83, 120°/0.8, 1.4652, 1.1478; 84, 128°/0.5, 1.4630, 1.0955; 80, 97°/0.04, 1.4609, 1.0681; 55, 140°/0.5, 1.5050, 1.1127; 82, 164-6°/0.7, 1.4922. At present the alternative [ABP(S)][O][P(O)AB] structure can not be excluded. Phosphites ABPCl react with H_2S in the presence of tertiary amines to give *thiophosphites* of the type *ABP(SH)*. Examples included the following (A = B, % yield, b.p./mm., n_D^{20}, d_{20} given): *EtO*, 73°/15, 1.4632, 1.0788; *BuO*, 95, 183-4°/18, 1.4628, —; and A = *Et*, B = *BuO*, 50, 107-8°/15, 1.4840, 0.9030.

P. Dreyfuss

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MICHALSKI, JAN

Reaction of free thiocyanogen with dialkyl phosphites. Jan Michalski and Jan Wieczorkowski (Inst. Technol. [illegible]). Roczniki Chem. 29, 127-[illegible] (English summary). — The reaction between free thiocyanogen and dialkyl phosphites is suggested to be $(RO)_2POH + (SCN)_2 \rightarrow (RO)_2P(O)(CNS)$ (I) + HSCN. Two possible structures (involving the N:C:S (A) and SC:N (B)) for I are considered. The observed MR is in better accord with A. Amines react with I to give dialkyl phosphoroamidates.

Morris Eisenberg

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MICHALSKY, J

CZECH

Some additional 4,4'-bis(aminoalkyl)-2,2'-bithiazoles. J. Michalský and J. Borkovec (Masarykova Univ., Brno, Czech.). Chem. Listy 48, 1872-3(1954); cf. C.A. 49, 9027g.—Condensation of o-$C_6H_4(CO)_2NCHMeCOCH_2Br$ (I) and o-$C_6H_4(CO)_2NCHMeCH_2COCH_2Br$ (II) with $(CSNH_2)_2$ gave the corresponding *4,4'-bis(1,2-phthalimidoethyl)-2,2'-bithiazole* (III), and *4,4'-bis(2-phthalimidopropyl)-2,2'-bithiazole* (IV), hydrolyzed to the *di-HCl salts of 4,4'-bis(1-aminoethyl)-2,2'-bithiazole* (V) and *4,4'-bis(2-aminopropyl)-2,2'-bithiazole* (VI). Treating 4.8 g. o-$C_6H_4(CO)_2NCHMeCOCHN_2$ in 15 ml. AcOH with 40% HBr to decoloration, and dilg. the soln. with 500 ml. ice water gave 4.6 g. I, m. 107° (from EtOH). Heating 2 g. I with 200 ml. EtOH and 400 mg. $(CSNH_2)_2$ 30 min. at 100° gave 1 g. III, m. 302-3° (from C_6H_6). Refluxing 400 mg. III in 300 ml. AcOH and 20 ml. concd. HCl 8 hrs., adding 20 ml. AcOH and 50 ml. HCl, refluxing the mixt. 16 hrs., evapg. *in vacuo*, removing the phthalic acid, evapg. the soln. to dryness, dissolving the residue in EtOH, and ppig. with Et_2O gave 170 mg. V, decompg. without melting above 320° (from $EtOH-Et_2O$); dipicrate, decomp. without m. above 310°. Similarly were prepd. IV (69%), m. 238-9° (from EtOH), and VI (63%), decompg. without melting above 295° [*dipicrate*, m. 286-8° (decompn.) (from H_2O)]. M. Hudlický

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MICHALSKY, J.; BORKOVEC, J.; AMBROZ, M.

"Aminoalkylquinoxalines. II.", P. 065, ([illegible], Vol. [illegible], No. 6, June 1954, Praha, Czech.)

SO: Monthly List of East European Accessions (EEAL), LC, Vol. 4, No. 3, March 1955, Uncl.

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MICHALSKI, JAN

Thionicotinic acid and dinicotinoyl disulfide. Jaroslaw Bryson and Jan Michalski (Inst. Technol., Warsaw). *Roczniki Chem.* 28, 601-3(1954)(English summary).—Nicotinic acid (30 g.) is added in portions to 51 g. $SOCl_2$. After the initial violent reaction, the mixt. is stirred on a steam bath 3 hrs., the excess halide is distd. off, and the residue, b. 153-5° (reduced pressure), is mixed with 300 ml. dry C_5H_5N, and H_2S passed through the stirred soln. at 0-4° for 7 hrs. The mixt. is filtered, the filtrate is evapd. to dryness, powdered, washed with Et_2O, MeOH, ice-H_2O, and again with Et_2O and MeOH. Recrystn. of the powder from MeOH yields 20.5 g. thionicotinic acid, m. 145-7°, which is oxidized with iodine to the disulfide, m. 88-9°. Chester Placek

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Michalski, Jan

POL

Organophosphorus compounds of sulfur and selenium. II. Synthesis of tetraalkylselenopyrophosphates. Jan Michalski and Jan Wieczorkowski (Higher Polytech. School, Łódź, Poland). *Roczniki Chem.* 28, 233-8(1954)(English summary); cf. *C.A.* 49, 3786a.—Tetraalkylselenopyrophosphate, $Se[PO(OR)_2]_2$ (I), were prepd. by adding with good stirring 0.1 mole dialkyl chlorophosphate (*C.A.* 49, 2306c) to 0.1 mole of dried and powd. $(EtO)_2P(O)SeNa$ (cf. Foss, *C.A.* 42, 2537c) in 100 ml. boiling anhyd. ether (Hg-sealed stirrer, drying tube). The mixt. was refluxed 90 min., dist. with 50 ml. C_6H_6 in a separatory funnel, washed with 50 ml. water, the top layer washed with 5% $NaHCO_3$ (about 50 ml.), H_2O, and dried over Na_2SO_4. Reaction products were distd. *in vacuo* after removing the solvents *in vacuo*. The following esters were prepd. [alkyls, % yield b.p. (uncor.), n^{20}_D, n^{25}_D, and d_{20} given]: tetra-Et (II), 68, $b_{0.05}$ 107°, 1.4638, 1.4618, 1.3403; tetra-Pr, 79, $b_{0.01}$ 106-7°, 1.4570, 1.4543, 1.2065; tetra-iso-Pr, 45, $b_{0.01}$ 93°, 1.4510, 1.4490, 1.1970; tetra-Bu, 89, $b_{0.02}$ 121° (with partial decompn.), 1.4558, 1.4540, 1.1395; di-Et di-Pr [from $ClPO(OEt)_2$ and $(PrO)_2P(O)SeNa$], 80, $b_{0.01}$ 111-12°, 1.4482, 1.4468, 1.2056. Prepn. of the tetra-Me ester was unsuccessful. I possess unpleasant odors, decomp. above 150°, are sparingly sol. in H_2O and easily sol. in organic solvents, and are less susceptible to hydrolysis than the corresponding O-derivs. I with concd. H_2SO_4 ppts. Se; they react with $AgNO_3$ and $HgCl_2$. The proposed structure requires further confirmation. II exhibits *anticholinesterase activity*.

Adam Sporzyński

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from $ClHMeCHCO_2Et$, a stoichiometric amt. of K, and 20% excess X, using procedure *A* (negligible heat effect), $b_{0.03}$ 98°, n^{25}_D 1.4458, d_{20} 1.1082. $(EtO)_2P(O)CH(CHPhCH_2CO_2Et)CO_2Et$, prepd. in 50% yield from X and V under the same conditions as XIV, $b_{0.02}$ 138°, n^{25}_D 1.4878, d_{20} 1.1397. $(EtO)_2P(O)CBu(CH_2CH_2CN)CO_2Et$ (XV) was prepd. in 73% yield from XI and VI (procedure *A*), using 0.1 mole K per mole of XI, $b_{0.03}$ 116–18°, n^{25}_D 1.4512, d_{20} 1.0631; $(EtO)_2P(O)CBu(CH_2CH_2CO_2Me)CO_2Et$ was prepd. in 73% yield from XI and VII, using procedure analogous to XV, $b_{0.05}$ 117–19°, n^{25}_D 1.4526, d_{20} 1.0973. $(EtO)_2P(O)C(CH_2CH_2CN)_2CN$ was prepd. from II and VI (procedure *B*). The crude product after the removal of the solvent was crystd. from CCl_4 and ether, yield 80%; colorless needles, m. 73.5–1°, easily sol. in C_6H_6, alc., difficulty in water, and petr. ether.

Adam Sporzyński

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and the mixt. was distd. as above, yielding 71 g. II, $b_{0.4}$ 124–8°, n^{20}_D 1.4370. VI (15.9 g.) was added (stirring, $CaCl_2$ tube) to a mixt. of 100 ml. C_6H_6, 0.69 g. Na, and 67.2 g. X kept at 25° (ice-water cooling), the mixt. heated 2 hrs. at 65°, cooled, neutralized with AcOH, washed with water, $NaHCO_3$ soln., and again water, dried over Na_2SO_4, the solvent removed, and the residue distd. *in vacuo* (procedure *A*), yielding the following fractions: Unchanged X (18 g.), $b_{0.05}$ 74–96°; $(EtO)_2P(O)CH(CH_2CH_2CN)CO_2Et$ (35 g.), $b_{0.05}$ 100–20°; 33 g. after redistn., $b_{0.05}$ 112–17°, n^{20}_D 1.4470, d_{20} 1.1168), and $(EtO)_2P(O)C(CH_2CH_2CN)_2CO_2Et$ (XIII) (19 g.), $b_{0.01-0.05}$ 118–60°. X (67.2 g.) was added (stirring, $CaCl_2$ tube) to 100 ml. dry C_6H_6 and 2.5 g. K (highly exothermic reaction), the mixt. cooled to room temp. and to it was added 31.8 g. VI at 45–50° (ice-water cooling), the mixt. then heated 3 hrs. at 60°, left 12 hrs. at room temp., and worked up as in *A*, yielding 68 g. XIII (procedure *B*), $b_{0.01}$ 145–7°, n^{20}_D 1.4630, d_{20} 1.1461. X (50% excess) treated with VII by procedure *A*, yielding 57% $(EtO)_2P(O)CH(CH_2CH_2CO_2Me)CO_2Et$, $b_{0.05}$ 96°, n^{20}_D 1.4448, d_{20} 1.1468. $(EtO)_2P(O)C(CH_2CH_2CO_2Me)_2CO_2Et$ was prepd. in 67% yield by procedure *B*, using 25% excess VII, $b_{0.05}$ 126–9°, n^{20}_D 1.4568, d_{20} 1.1884. $(EtO)_2P(O)CH(CHMeCH_2CO_2Et)CO_2Et$ (XIV) was prepd. in 66% yield

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in vacuo through a short Vigreux column, yielding 152 g. $(EtO)_2P(O)CH_2CO_2Et$ (X), b_{12} 147-9°, n_D^{20} 1.4305 (all b.ps. and m.ps. uncorr.). X was added with stirring (Hg-sealed stirrer) to a mixt. of 13 g. K in 200 ml. xylene (exothermic reaction), the mixt. cooled and to it was added 47 g. BuBr, the ppt. filtered off, the filtrate washed with 1% HCl, H_2O, 5% $NaHCO_3$, and H_2O, the xylene layer dried over Na_2SO_4, the xylene removed, and the residue distd. as above, yielding 62 g. $(EtO)_2P(O)CHBuCO_2Et$ (XI), b_{12} 121-2°, n_D^{20} 1.4358. $P(OEt)_3$ (XII) (40 g.) and 58 g. $EtCHBrCO_2Et$ were refluxed 4 hrs. on the oil bath at 165° and the mixt. was distd. as above, yielding 52 g. $(EtO)_2P(O)CHEtCO_2Et$, b_{12} 152-4°, n_D^{20} 1.4290. To the suspension of 9.2 g. Na in 200 ml. xylene was added 55.2 g. VIII followed by 80 g. $MeCHBrCO_2Et$, the mixt. refluxed 3 hrs. at 60°, the ppt. filtered off, and the reaction product worked up as above, yielding 65 g. $(EtO)_2P(O)CHMeCO_2Et$, b_{12} 150-1°, n_D^{20} 1.4282. XII (88.8 g.) and 40 g. $ClCH_2CN$ were refluxed 4 hrs. in the oil bath at 165-70°,

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MICHALSKI, JAN

POL

Synthesis of organophosphorus compounds based on phosphonoacetic ester and its analogs. Addition of phosphonoacetic ester, alkylated phosphonoacetic esters, and phosphonoacetic nitrile to α,β-unsaturated esters and nitriles. Bernard Fitzner and Jan Michalski (Higher Polytech. School, Łódź, Poland). *Chemia Stosow.* 28, 185-95 (1954) (English summary). $(EtO)_2P(O)CH_2CO_2Et$ (I), $(Et-O)_2P(O)CH_2CN$ (II), and alkylated I (III) in the presence of alk. catalysts undergo Michael condensation with α,β-unsatd. esters and nitriles at 20-80° in C_6H_6 or PhMe (less satisfactory results are obtained in alc. soln.). III are less reactive than I or II while the order of decreasing reactivity of α,β-unsatd. compounds (IV) is: acrylates > crotonates, methacrylates > $PhCH:CHCO_2Et$ (V). Condensation of active reagents (e.g., I with $CH_2:CHCN$ (VI) or $CH_2:CHCO_2Me$ (VII)) is exothermic, requires small amts. of catalyst, and yields a mixt. of mono- and disubstituted derivs. Condensation of a reactive P compd. with less reactive IV requires heating, more catalyst and can be stopped at the first stage, while the poorly reactive compounds do not condense even under drastic conditions due to steric hindrance. A mechanism of condensation is given. To 23 g. Na finely dispersed in 400 ml. dry xylene was added with cooling and stirring (Hg-sealed stirrer) 138 g. $HP(O)(OEt)_2$ (VIII), followed by 150 g. $ClCH_2CO_2Et$ (IX), the mixt. heated 6 hrs. on the bath at 60°, NaCl centrifuged off, the xylene removed, and the residue distd.

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MICHALSKY, Jiri

Preparation of 1,4-diamino-2-butanone. J. Michálský, J. Borkovec, and [illegible] (Masarykova Univ., Brno, Czech.). Chem. Listy 47, 1239-41(1953).—H_2NCH_2CO-$CH_2CH_2NH_2$ (I) was prepd. by the following series of reactions: o-$C_6H_4(CO)_2NCH_2COCl$ and CH_2N_2 gave 96% o-$C_6H_4(CO)_2NCH_2COCHN_2$, m. 167-8° (decompn.) (from Et_2O or dioxane), which was transformed in 66% yield to o-$C_6H_4(CO)_2NCH_2CH_2CO_2Me$, m. 71-2° (from Et_2O). This was hydrolyzed by heating 20 min. at 80° with aq. HBr (d. 1.38) to 84% o-$C_6H_4(CO)_2NCH_2CH_2CO_2H$, m. 148-9° (from C_6H_6), which treated with $SOCl_2$ and then with CH_2N_2 yielded 78% o-$C_6H_4(CO)_2NCH_2CH_2COCHN_2$ (II), m. 122° (from Et_2O). Treatment of II with HCl in Et_2O gave 88.7% yield of o-$C_6H_4(CO)_2NCH_2CH_2COCH_2Cl$; this heated with o-$C_6H_4(CO)_2NK$ 6 hrs. on the steam bath yielded 52% of *1,4-diphthalimido-2-butanone*, m. 248-9°, which refluxed 36 hrs. with 37% HCl in AcOH (1:1) gave 78% I, m. 220-1° (decompn.). M. Hudlický

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BERNARD FISZER

$(iso\text{-}BuO)_2P(O)SPO(OBu\text{-}iso)_2$ (XI), colorless liquid, $b_{0.5}$ 96-8°, d_{20} = 1.0488, n_D^{20} = 1.4463; $(MeO)_2POCl$ (below 5°) gave a sirupy, malodorous liquid, insol. in org. solvents, sol. in water. Et_3N (0.25 mole) added dropwise to 0.2 mole di-Et phosphite and 0.6 mole CCl_4 and treated with H_2S with stirring at 5-10° for 3 hrs. gave 65% III. All boiling points are uncor. Tetraalkyl thiopyrophosphates are insecticides. The following doses (in g./kg. wt. of animal) were fatal to mice and rats when injected intramuscularly: III, 0.000055; V and X, 0.00025; VI, 0.001; VIII, 0.0025; IX and XI, 0.05. All compds. stopped cholinesterase activity in rat brain, III being the most active and approaching parathion in effectiveness. J. R. Spencer

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BERNARD FISZER

P(O)SNa (IV) and $(MeO)_2POCl$ gave 21% $(MeO)_2P(O)SP(O)(OMe)_2$ (V), yellow liquid, $b_{0.01}$ 128–30° (decompn. slightly), $b_{0.02}$ 68–9° (bath at 100°), d_{25} = 1.3360, n_D^{25} = 1.4519. IV and II gave 50% $(MeO)_2P(O)SP(O)(OEt)_2$ (VI), colorless liquid, $b_{0.01}$ 116°, d_{25} = 1.2530, n_D^{25} = 1.4409. I and $(BuO)_2POCl$ gave 57 g. crude product which in a mol. distn. (at 0.001 mm., condenser at 20 mm. from liquid surface, distg. at 5 drops/min.) gave the following fractions: (1).—6 g., temp. of liquid 50–88°, n_D^{25} = 1.4398; (2).—13.5 g., temp. of liquid 88°, n_D^{25} = 1.4471, 16.8% P [calcd. for $(BuO)_2P(O)SP(O)(OEt)_2$ 17.1% P]; (3).—27.5 g., temp. of liquid 97°, n_D^{25} = 1.4415, 14.5% P; (4).—2.5 g., temp. of liquid 117°, n_D^{25} = 1.4340. I and $(PrO)_2POCl$ (VII) gave 77% $(PrO)_2P(O)SP(O)(OEt)_2$, colorless liquid, $b_{0.001}$ 103.5°, d_{25} = 1.1347, n_D^{25} = 1.4429. II (34.5 g., 0.2 mole) was added dropwise with stirring at 10–15° to 70 ml. dry pyridine and a strong stream of H_2S was passed through the stirred and cooled mixt. for 90 min. The pptd. $C_5H_5N.HCl$ was filtered and washed twice with 75 ml. C_6H_6. The combined filtrates were distd. under reduced pressure and the residue taken up in 100 ml. C_6H_6, washed successively with 30 ml. 2% HCl, 30 ml. water, 30 ml. 5% $NaHCO_3$, and 30 ml. water, dried with Na_2SO_4, and distd. twice gave 22 g. (74%) III, $b_{0.01}$ 82–4°. An 85% yield of III was obtained when *N*-methylmorpholine (dild. with C_6H_6) was used instead of pyridine. Similarly, VII and H_2S in pyridine gave 67% $(PrO)_2P(O)SP(O)(OPr)_2$ (VIII), colorless liquid, $b_{0.01}$ 94–5°, d_{25} = 1.1075, n_D^{25} = 1.4363; (iso-PrO)$_2$POCl gave 70% (iso-PrO)$_2$P(O)SPO(OPr-iso)$_2$ (IX), colorless liquid, $b_{0.01}$ 82–4°, d_{25} = 1.0885, n_D^{25} = 1.4370; $(BuO)_2POCl$ gave 72% $(BuO)_2POSPO(OBu)_2$ (X), yellow liquid, $b_{0.01}$ 112–14°, d_{25} = 1.0374, n_D^{25} = 1.4517; (iso-BuO)$_2$POCl gave 72%

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MICHALSKI, JAN POL.

Organophosphorus compounds of sulfur and selenium. I. Synthesis of tetraalkyl thiopyrophosphates. [illegible] Fiszer, Jan Michalski, and Jan Wieczorkowski (Polytech. Łódź, Poland). Roczniki Chem. 27, 482-93(1953)(English summary).—Tetraalkyl thiopyrophosphates, $(RO)_2P(O)SP(O)(OR')_2$, may be prepd. by: (1).—condensation of Na O,O-dialkylthiophosphates with dialkyl chlorophosphates or (2).—action of H_2S on dialkyl chlorophosphates in the presence of tertiary amines. Toxicity and anticholinesterase activity of the products were studied. Dialkyl chlorophosphates were prepd. using the method described previously (cf. C.A. 49, 2306c). Na O,O-dialkylthiophosphates were obtained by adding 0.25 mole powd. S in small portions with stirring and outside cooling to a mixt. of 0.2 mole Na dissolved in 60 ml. dry ROH and 0.21 mole dialkyl phosphite in 80 ml. dry Et_2O. Stirring was continued after all the S was added until the mixt. warmed up to room temp. The excess S was filtered and the filtrate was evapd. under reduced pressure at room temp. The cryst. product was washed 3 times with Et_2O and evapd. each time. Di-Et phosphite gave 98% yield (based on Na) crude $(EtO)_2P(O)SNa$ (I), m. 188° [m. 203° (from $CHCl_3$-Et_2O)]. Crude I was used in further syntheses. $(EtO)_2POCl$ (II) (21.5 g.) (0.125 mole) added dropwise with vigorous stirring to a refluxing mixt. of 24 g. (0.125 mole) powd. dry I and 120 ml. anhyd. Et_2O, refluxed for 30 min., dild. with 100 ml. C_6H_6, washed successively with: 100 ml. water contg. a few drops pyridine, 50 ml. 1% HCl, 50 ml. water, 50 ml. 5% $NaHCO_3$, and 50 ml. water, dried with Na_2SO_4 and distd. twice gave 20 g. (65%) $(EtO)_2P(O)SP(O)(OEt)_2$ (III), $b_{0.5}$ 120-2°, d_{20} = 1.1855, n_D^{20} = 1.4400. Similarly, $(MeO)_2$-

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[illegible]

Simplified method for preparation of dialkyl chlorophosphonates. H. Fiszer and J. Michalski (Rhone, Chem. 1957, 26, 683–689).—The method [illegible] treating 1.5 mol. of dry benzene [illegible] equal volume of a suitable alcohol with 0.5 mol. of PCl_3 (diluted with 0.5 mol. of benzene) and 0.5 mol. SO_2Cl_2 (diluted with 0.5 mol. of benzene) at 8–10°. The gaseous reaction products, alkyl chloride and the solvent, are distilled off at room temp. under reduced pressure, and the dialkyl chlorophosphonate formed is purified by vac. distillation. The reaction time is considerably shortened and the yield increased by about 10% by using this method (~90% yield for Et_2, Pr^{n}_2, Pr^{i}_2, Bu^{n}_2, and Bu^{i}_2 esters). A. STORFER.

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MICHALSKI, J.

Chemical Abst.
Vol. 48 No. 9
May 10, 1954
Organic Chemistry

Tetraalkyl thiopyrophosphates. B. Fizer and J. Michalski (Politech., Lodz, Poland). *Roczniki Chem.* 25, 614-16(1951)(English summary).—Tetraalkyl thiopyrophosphates of the thioanhydride type were prepd.: (*A*) by condensation of the corresponding dialkyl chlorophosphonates with salts of dialkyl thiophosphoric acids according to $(RO)_2P(:O)SNa + Cl(O:)P(OR)_2 \rightarrow (RO)_2P(:O)S(O:)P(OR)_2 + NaCl$, and (*B*) by action of H_2S on dialkyl chlorophosphonates in the presence of a tertiary base (e.g. C_5H_5N) according to $2(RO)_2P(:O)Cl + H_2S + 2C_5H_5N \rightarrow (RO)_2P(:O)S(O:)P(OR)_2 + 2C_5H_5N.HCl$. The following *thiopyrophosphates* were prepd.: *tetra-Et*, $b_{0.4}$ 120-2°, (74%); *tetra-iso-Pr*, $b_{0.01}$ 82-4° (66%), *tetra-Bu*, $b_{0.01}$ 112-14° (72%), and *tetra-iso-Bu*, $b_{0.01}$ 96-8° (72%). G. A. W.

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MICHALSKI JAN

5

Organic phosphorus compounds. I. Addition of dialkyl hydrogen phosphonates to ethylenic derivatives. Bolesław Bochwic and Jan Michalski (Inst. Technol., Łódź, Poland). *Roczniki Chem.* 25, 396-40(1951)(English summary); cf. *C.A.* 46, 8857.—A new method for the synthesis of phosphonic acids is described; it is based on the addn. reaction of dialkyl H phosphonates to unsatd. compds. contg. activating groups (CO, CO_2R, CN) in α-position to the ethylenic bond. To 19.4 g. $HPO(OBu)_2$ (I) in 20 ml. C_6H_6 in a flask with stirrer, dropping funnel, thermometer, and $CaCl_2$ tube, was added 0.15 g. Na; the flask cooled to 10°, 13.7 g. $MeCH:CHCO_2Me$ in 25 ml. C_6H_6 added dropwise at 15-20°, the mixt. let stand 24 hrs., the brown product dissolved in 160 ml. C_6H_6, the soln. washed with a NaCl soln. in 0.1*N* HCl, and then NaCl in water, the C_6H_6 layer dried with $MgSO_4$, and dark brown product distd. *in vacuo*, giving 23.3 g. (62%) *Me 3-(dibutylphosphono)butyrate* (II), $b_{0.05}$ 83-4°. Hydrolysis of 5 g. II with 25 ml. 20% HCl gave 3-phosphonobutyric acid (III), colorless needles from glacial AcOH, m. 149-51°, sol. in H_2O, alc., and Me_2CO, insol. in ether, C_6H_6, and ligroine. II was also obtained from $MeCHBrCH_2CO_2Et$ (IV): a suspension of 1.02 g. Na in 50 ml. boiling PhMe in the app. described above, cooled to 70°, 9 g. I added dropwise at a temp. up to 80°, the flask warmed in a water bath 2 hrs., 9 g. IV then added dropwise at 60-70°, the flask warmed another 2 hrs., the cooled product washed with satd. NaCl, and the PhMe layer dried with $MgSO_4$ and distd. *in vacuo* gave 8.3 g. (62%) II, b. 76-9°/0.001 mm., n_D^{20} 1.4381. Na (0.15 g.) in a mixt. of 19.4 g. I and 20 ml. C_6H_6 treated dropwise with 17.2 g. $CH_2:CHCO_2Me$ at 10° (exothermic reaction) with the temp. kept at 20-5°, and the product, *Me 3-(dibutylphosphono)propionate* (V), distd. under 0.001 mm. Hg gave 3 fractions: to 78° (3.0 g.), 78-90° (10.5 g.), 90-130° (7.0 g.), n_D^{20} 1.4365, 1.4383, 1.4453, resp.; all 3 fractions upon hydrolysis with 20% HCl gave cryst. $(HO)_2P(O)CH_2CH_2CO_2H$ (VI). $PhCH:CHCO_2Et$ (26.4 g.) added dropwise at 15-20° to 19.4 g. I in 20 ml. C_6H_6 contg. 0.15 g. Na gave 15 g. (40%) *Et 3-(dibutylphosphono)-3-phenylpropionate* (VII), $b_{0.05}$ 123°. Hydrolysis of 5.0 g. VII yielded 2.9 g. 3-phosphono-3-phenylpropionic acid (VIII), colorless needles from glacial AcOH, m. 199-202°. Similarly, 19.4 g. I in 20 ml. C_6H_6 and 0.15 g. Na with 21.7 g. $PhCH:CHAc$ in 15 ml. C_6H_6 at 15-20° gave 23 g. (68 g.) *1-phenyl-1-(dibutylphosphono)-3-butanone* (IX), $b_{0.05}$ 117°. Hydrolysis of 10 g. IX with 30 ml. 20% HCl in 10 hrs. gave 6.1 g. (90%) crude keto acid (X), crystn. of which failed. X (0.1 g.) in 1 ml. MeOH treated with Brady reagent [$2,4-(O_2N)_2C_6H_3NHNH_2$; cf. *C.A.* 25, 3310] gave the 2,4-dinitrophenylhydrazone (XI), yellow needles, from aq. MeOH, m. 103-4°. XI (5.8 g.) in 50 ml. H_2O added dropwise to 6.5 g. NaOH, 12 g. Br, and 200 ml. H_2O, the mixt. heated 1 hr. at 70°, acidified with 20 g. 30% HCl, 3 g. Na_2SO_3 added, the soln. evapd., the residue dried, extd. with 300 ml. EtOH, the ext. evapd., and the cryst. residue recrystd. from glacial AcOH gave VIII, m. 198-201°. II (26.2 g.) in 50 ml. C_6H_6 and 0.2 g. Na treated dropwise with 11.0 g. $CH_2:CHCN$ at 20-5° (exothermic reaction) gave 27 g. (84%) *di-Bu (β-cyanoethyl)phosphonate* (XII), m. 143-4°/0.8 mm. XII (5.0 g.) hydrolyzed 10 hrs. with 25 ml. 20% HCl, evapd., and the residue dried over KOH, extd. with 100 ml. abs. EtOH, and recrystd. from H_2O yielded 2.7 g. (87%) 3-phosphonopropionic acid, m. 107-8°.

Gene A. Wozny

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CA MICHALSKI, J

10

Cholic acids. J. Hadáček and J. Michalský. *Časopis Českého Lékarnictva* 62, 120-5(1949). Expts. were made to extend the side chain of 3α-hydroxy-7,12-diketocholic acid by the prepn. of the acetoxy-, diacetoxy-, triketo-, chloro-, and amide compds. The syntheses were verified by elemental analyses. Oldrich Sebek

1952

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MICHALSKI, Jacek

Biology of Scolytus (Scolytochalus) ensifer Eichh. (Coleoptera, Scolytidae). Trace mark role. Lesn 16 no.3:31-72 '63.

1. Department of Forest Protection of the School of Agriculture, Poznan.

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BALAZY, Stanislaw; MICHALSKI, Jacek

The parasitic Hymenoptera of the (Coleoptera, Scolytidae) occurring in Poland. Prace nauk roln i lesn 13 no.1:71-141 '62.

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MICHALSKI, Jacek

Natural enemies of Scolytus (Scolytochelus) ensifer Eichh. (Coleoptera, Scolytidae). Prace nauk roln i lesn 13 no.1:15-49 '62.

1. Katedra Ochrony Lasu, Wyzsza Szkola Rolnicza, Poznan.

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MICHALKSI, Jacek
SZMIDT, A.

Observations on some methods of fighting Blastophaga. p. 55

SYLWAN. (Wydzial Nauk Rolniczych i Lesnych Polskiej Akademii Nauk i Polskie Towarzystwo Lesne) Warszawa, Poland
Vol. 101, no. 7, July 1957

Monthly List of East European Accessions Index (EEAI), LC, Vol. 8, no. 6, June 1959
Uncl.

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8

Author : Michalski, J.
Inst :
Title : A New Species in the Fauna of Poland - Scolytus ensifer Eichh. (Coleoptera, Scolytidae).

Orig Pub : Polskie pismo entomol., 1956, (1957), 26, No 1-26, 461.

Abstract : No abstract.

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MIKHAL'SKIY, Ya. [Michalski, J.]

New palaearctic species of [illegible] beetles of the genus Scolytus Geoffr (Coleoptera, Scolytidae). Ent. obozr. 23 no.3:[illegible] '[illegible]. (MIRA 17:10)

1. Vysshaya shkola sel'skogo khozyaystva, Lesnoy fakul'tet, kafedra zashchity lesa, Poznan', Pol'sha.

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MICHALSKI, I.

SOURCE

periodicals: BULLETIN Vol. 9, no. [illegible], 1958. In English.

MICHALSKI, I. From the studies in the anthropological structure of the German nation; northwestern Germany. p. 1.

Monthly List of East European Accessions (EEAI) LC Vol. [illegible], no. 5 May 1959, Unclass.

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MICHALSKI, I,

GEOGRAPHY & GEOLOGY

PERIODICAL: CZLOWIEK W CZASIE I PRZESTRZENI. Vol 1, no. 4, 1958

MICHALSKI, I. Anthropological types in Egypt, p. 192

Monthly List of East European Accessions (EEAI) LC. Vol 8, no. 5, May 1959, Unclass.

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ACC NR: AM7002488

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Territorial defense considered within the defense system of a country -- 77
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ACC NR: ~~AM5028974~~
AM7002488

Monograph

PO/

Michalski, Henryk

Territorial defense strategy and tactics (Strategia i taktyka obrony terytorium kraju) [Warsaw; Wyd-wo MON] 1965. 451 p. illus., biblio. Errata slip inserted. 1,500 copies printed. Series note: Biblioteka Polskiej mysli wojskowej.

TOPIC TAGS: civil defense, air defense system, military operation, ground force tactic

PURPOSE AND COVERAGE: This book, published by the publishing house of the Ministry of National Defense, Warsaw, is intended for the general reader. Based on Western, Polish, and Soviet sources, it presents a general survey of the modern territorial defense system of a country, with emphasis laid on the defensive needs of Poland. Particular attention is given to the role of civilians in territorial defense. The text includes 18 diagrams and other tables and illustrative material.

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Some history up to 1945 -- 13

Ominous implications of new weapons -- 43

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H

COUNTRY : POLAND
CATEGORY : Chemical Technology. Chemical Products and Their Applications. Chemical Engineering
ABS. JOUR. : RZKhim., No. 23 1959, No. 82521

AUTHOR : Michalski, H.; Strumillo, G.; Szapiro, S.
INST. : -
TITLE : Determination Method of Packing Height in Rectifying Columns

ORIG. PUB. : Zesz. nauk. Politechn. lodzkiej, 1957, No 16, 3-14

ABSTRACT : A method for determination packing height is proposed which is based on the results of experiments conducted by the authors on a laboratory installation and suitable for calculations of commercial rectifying columns. A satisfactory correlation of data is established when employing factors obtained by the proposed method as well as data found by the others. A satisfactory correlation exists also with data obtained by the authors in their experiments with different packed columns.

CARD: 1/1

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MICHALSKI, Franciszek

On the programme of complex wood treatment. Pt. 4. Przem drzew 13 no.1:4-7 Ja '62.

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Franciszek

The program of complex wood treatment. Pt.3. (To be contd.) Przem drzew 12 no.12:1-3 '61.

(Wood)

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Ireneusz

Taxonomic and populationistic trends in modern anthropology; some remarks in connection with Tadeusz Bielicki's article: "The Anthropological Controversy on the Concept of Race." Kosmos biol 12 no.1: 51-78 '63.

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Henryk; SERWINSKI, Mieczyslaw

Determination of the mass-transfer coefficient in a distillation wetted-wall column. Rocz tech chem zywn 8:87-97 '61.

1. Department of Chemical Engineering, Institute of Technology, Lodz.

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

KIRSCHNER, Henryk; MICHALSKI, Ewald

The value of determining the physical capacity based on a brief effort test. Med. pracy 16 no.2:113-123 '65

1. Z Zakładu Fizjologii Pracy Akademii Medycznej w Warszawie (Kierownik [illegible]

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

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ASKANAS, Zdzislaw; CZERWINSKA, Stanislawa; LISZEWSKA, Danuta; MICHALSKI, Eugeniusz; RUDNICKI, Stanislaw; RYWIK, Stefan; SLIDZIEWSKI, Konstanty

A method for the selection of a representative sample for the investigation of the level of arterial pressure in large population groups. Pol. tyg. lek. 20 no.23:830-834 7 Je '65.

1. Z Centralnej Przychodni Chorob Ukladu Krazenia w Warszawie (Kierownik: prof. dr. med. Zdzislaw Askanas).

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

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MICHALSKI, Eugeniusz

POLAND

MICHALSKI, Eugeniusz; CZARNECKI, Kazimierz; PIETRUCHA, Krystyna

Department of Inorganic Chemistry, University of Lodz
(Zaklad Chemii Nieorganicznej Uniwersytetu, Lodz)

Warsaw, Chemia analityczna, No 5, 1963, pp 713-17.

"Catalytic Microdetermination of Ferrous Ions from Amperometric Measurements".

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Eugeniusz; GELOWA, Helena

Catalytic determination of microgram amounts of germanium on the basis of amperometric measurements. Chem anal 8 no.4:643 '63.

1. Department of Inorganic Chemistry, University, Lodz.

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Eugeniusz; WTORKOWSKA, Albina

Catalytic determination of trace amounts of thiosulfate ions by amperometric measurements. Chem anal 7 no.4:783-790 '62.

1. Department of Inorganic Chemistry, University, Lodz.

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

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BIALECKI, S.; BOJKO, M.; JOZEFACIUK, D.; LESZEK, H.; MICHALSKI, E.; RUSZCZYNSKA, J.; SARNECKA, D.; WOJCIECHOWSKI, J.

Causes of delayed union and pseudarthrosis of the long bone. Chir. narz. ruchu ortop. polska 26 no.5:597-604 '61.

1. Z Kliniki Ortopedycznej AM w Warszawie Kierownik: prof. dr. A.Gruca.

(FRACTURES UNUNITED etiol) (PSEUDARTHROSIS etiol)

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Eugeniusz; PAWLUK, Natalia

Amperometric Red-ox titrations without external voltage. II. Titration of reversible systems by reversible titrants. Chem anal 6 no.6:943-948 '61.

1. Department of Inorganic Chemistry, University, Lodz.

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Eugeniusz; WTORKOWSKA, Albina

Catalytic ultramicrodetermination of thiocyanide on the basis of amperometric measurements. Chem anal 6 no.3:365-375 '61.

1. Department of Inorganic Chemistry, University, Lodz.

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Eugeniusz; WALEWSKI, Ludwik

Catalytic microdetermination of phosphate by means of amperometry.
Chem anal 6 no.2:273 '61. (EEAI 10:9)

1. Department of Inorganic Chemistry, University, Lodz.

(Phosphates) (Conductometric analysis)

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Eugeniusz, prof. dr.

Amperometric analysis and its development in recent years. Wiad chem 14 no.7:411-423 Jl '60.

1. Kierownik Katedra Chemii Nieorganicznej, Uniwersytet, Lodz.

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

MICHALSKI, Eugeniusz; TUROWSKA, Maria

Analytical application of chemiluminescent diacridine derivatives. I. Identification of some aliphatic alcohols. II. Determination of methanol and ethanol in their mixture. Chem anal 5 no.4:625-636 '60. (EEAI 10:9)

1. Department of Inorganic Chemistry, University, Lodz.

(Biacridine) (Luminescence) (Aliphatic compounds)
(Alcohols) (Methanol) (Ethyl alcohol)

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

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MICHALSKI, E. ; CZARNICKI, K.

Amperometric determination of some organic acids without additional voltage from outside. p. 83.

CHEMIA ANALITYCZNA. Warszawa, Poland. No. 8, August 1959.

Monthly List of East European Accessions (EEAI) LC, Vol. 8, No. 11 November 1959.

Uncl.

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E-1

COUNTRY : Poland
CATEGORY :

78256

ABS. JOUR. : RZKhim., No. 22 1959, No.

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : and phenolphthalein as indicators. The relative differences in the results obtained from titrations of HCl solutions with NaOH solutions using CI are 0.12% when compared with the results obtained when bromthymol blue is used and 0.05% when phenolphthalein is used as the comparison standard. The addition of aliphatic alconols (methyl, ethyl, n-propyl, isobutyl) to the solutions to be titrated increases the intensity of luminescence and the accuracy of the titration.

CARD: 3/4

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E-1

COUNTRY : Poland
CATEGORY :

ABS. JOUR. : RZKhim., No. 22 1959, No. 78256

AUTHOR :
INST. :
TITLE :

ORIG. PUB. :

ABSTRACT : of the titration is determined by the appearance of yellow-green chemiluminescence throughout the solution on the addition of 1 drop of NaOH; in the titration of NaOH solutions with HCl solutions the endpoint is determined by the disappearance of the yellow-green chemiluminescence. Potentiometric measurements have shown that the indicator color change takes place at pH 8.6 for I, 8.5 for II, 8.2 for III, and 8.1 for IV. In order to determine the accuracy of the titrations, duplicate titrations were made using bromthymol blue

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APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

E-1

COUNTRY : Poland
CATEGORY : Analytical Chemistry--General

78256

ABS. JOUR. : RZKhim., No. 22 1959, No.

AUTHOR : Michalski, E. and Turowska, M.
INST. : Not given
TITLE : Derivatives of Diacridine as Chemiluminescent Indicators. I.

ORIG. PUB. : Chem Analit, 3, No 3-4, 599-607 (1958)

ABSTRACT : The feasibility of the application of the nitrates of N,N'-dipropyl- (I), N,N'-ditolyl- (II), N,N-diphenyl- (III), and N,N-diallyldiacridine (IV) as chemiluminescent indicators (CI) in the titration of strong acids with strong bases has been investigated. 0.01 N, 0.1 N, and 0.5 N solutions of HCl and NaOH were used in the titrations. To the solutions to be titrated are added 5 ml of a 0.04% solution of CI and 5 ml of 3% H_2O_2; the resulting solution is titrated in the dark (magnetic stirring) with CO_2-free NaOH. The endpoint

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E-2

COUNTRY : Poland
CATEGORY :

ABS. JOUR. : RZKhim., No. 1959, No. 86100

AUTHOR : Michalski, E.; Stapor, W.
INST. :
TITLE : Amperometric Titration of Thallium without Application of External Voltage

ORIG. PUB. : Chem. analit., 1958, 3, No 3-4, 441-444

ABSTRACT : Description of amperometric titration of Tl^+ with a solution of KI, in a medium of 20% C_2H_5OH, without application of external voltage, using a stationary and a rotating Pt-electrode. As the comparison electrode is used a calomel electrode in which saturated KCl-solution is replaced by a solution of KNO_3; the potential of such an electrode is close the potential of indicator Pt-electrode at equivalence point. Best results were obtained on using a rotating Pt-electrode. By the described method, up to 40 mg Tl^+ can be determined with an error of 0.1%.
V. Mirkin.

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5

Amperometric determination of calcium with the aid of one and two indicator electrodes. Bogusław Michalski and Zbigniew Gulus (Univ. Łódź, Poland). Chem. Anal. (Warsaw) 3, 431-8(1958)(English summary).—A method for detg. Ca and Ca + Mg in limestone with the aid of one and two indicator electrodes is described. The end point of titration was detd.: (a) amperometrically without supplying any external e.m.f.; the elec. system consisted of an indicator electrode made of Pt (4 mm. long and 0.5 mm. in diam.) and a reference electrode Hg/Hg_2SO_4, N H_2SO_4 connected to a galvanometer with a sensitivity of 8×10^{-9} amp./mm./m. and (b) by dead stop method with 2 indicator electrodes. *Determination of calcium.* To two 20 ml. neutral samples contg. 60-100 mg. Ca, add 4 g. NH_4Cl; after dissolution add to one of them 96 ml. 96% EtOH and titrate with 0.2N $K_4Fe(CN)_6$ (preliminary titration). To the other add EtOH in amts. calcd. from the preliminary titration $A = (20 + a)2.53$, a being vol. $K_4Fe(CN)_6$ in ml. Titrate the sample with 0.2N $K_4Fe(CN)_6$. A series of Ca analyses contg. about 60, 80 and 100 mg. were made. The mean error of detn. varied from 0.44 to −0.46%. Titration of 0.2N Mg soln. with $K_4Fe(CN)_6$ gave results about 1% higher than theoretical. *Determination of Ca + Mg in limestone.* To about 5.2 g. limestone add HCl and heat to dissolution. Dil. the soln. contg. SiO_2 with H_2O to 200 ml., add a few drops of methyl orange, and dropwise NH_3 with stirring. Filter and wash ppt. with NH_4Cl soln., redissolve, and reppt. with NH_3. Add to the filtrates H_2O to make 500 ml. Further procedure as above. The contents of Ca in limestone were: 38.77, 37.1, and 37.8%; those of Ca + Mg, recalcd. to CaO, detd. by amperometric, complexometric, and gravimetric methods were: 54.88, 55.13, 55.03; 53.85, 53.92, 53.77; 54.23%, 53.38% and 54.43%.

Z. Kurtyka

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COUNTRY : Poland E-1
CATEGORY :

ABS. JOUR. : RZKhim., No. 1959, No. 86010

AUTHOR : Michalski, E.
INST. :
TITLE : Amperometric Titration without Application of External Voltage

ORIG. PUB. : Chem. analit., 1958, 3, No 3-4, 423-430

ABSTRACT : It is shown that there is considerable similarity between amperometric and potentiometric titration. In particular, amperometric titration without application of external voltage is analogous to classical potentiometric titration; in the 1st method is used one indicator electrode which can be, depending upon choice of comparison electrode, the cathode or the anode, while the titration reagent plays the role of depolarizer (polarizer) of indicator electrode. The theory of amperometric titration without application of external voltage, is described. At the author's laboratory methods have been developed for determination of I^-, Ag^+, Hg^{2+}, Hg_2^{2+}, Bi^{3+}, Tl^+, Ca^{2+}, and also of oxalic, tartaric, and citric acid. -- V. Mirkin.

CARD:

75

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MICHALSKI, E.

Preparation of pure crystalline sodium hydrogen sulfide. Eugeniusz Michalski, Ryszard Grochowski, and Zbigniew Galus (Univ. Łódź, Poland). *Zeszyty Nauk. Uniw. Łódź, Ser. II, Nauki Mat.-Przyrod.* No. 3, 125-32(1957)(English and Russian summaries).—$Ca(OH)_2$ (120 g.) of 70% purity was added to a suspension of 1 kg. Na_2S of 61% purity in 3 kg. H_2O. The mixt. at 95° was satd. with H_2S flowing at 16 l./hr. and was cooled to 50° when the Na_2S was dissolved. H_2S addn. was stopped when the pH became nearly const. Pale yellow $NaHS.3H_2O$ contg. 50.8% NaHS crystd. at 27°.

J. Stecki

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POLAND / Analytical Chemistry. Analysis of Inorganic Substances. E-2

Abs Jour: Ref Zhur-Khimiya, No 1, 1959, 969.

Abstract: cipitate which formed had settled, the solution is filtered by suction through a sintered glass funnel and the precipitate of BiOI is washed twice with a small amount of water and dissolved in 18-40 ml of 6% HCl (depending on the amount of precipitate). The solution obtained is diluted with water to approximately 150 ml and while stirring vigorously is titrated amperometrically with 0.01-0.1 N solution of silver nitrate. The end point is determined graphically from a curve which expresses the relationship between current strength and the volume of the silver nitrate solution added, or directly from the zero location of the galvanometer indicator. The amperometric titration without external voltage is conducted by using an indicating Pt electrode which is placed into the

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APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

POLAND / Analytical Chemistry. Analysis of Inorganic Substances. E-2

Abs Jour: Ref Zhur-Khimiya, No 1, 1959, 969.

Author : Michalski, E., Ruskul, W.
Inst : Not given.
Title : The Amperometric Titration of Bismuth Without Using External Current.

Orig Pub: Chem analit., 1957, 2, No 3, 284-285.

Abstract: A description is given on a method which is based on a previous precipitation of Bi in the form BiOI, the precipitate obtained is dissolved in HCl and the iodine is then titrated ampometrically with silver nitrate solution, without using any external current. Potassium iodide is added (in a small excess) to the solution being analyzed, the mixture is diluted with hot water and a solution of sodium acetate is added. After the pre-

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[illegible] N,N'-Diphenyl- [illegible] N,N'-ditolyl [illegible] N,N'-diphenyl [illegible] as chemiluminescent indicators [illegible] (I), N,N'-Diphenyl- (II), N,N'-ditolyl (III) and N,N'-diphenylacridinium nitrate (IV) were investigated as acidobasic chemiluminescent indicators for the titration of strong acids by strong bases and of weak acids by strong bases. The catalytic influence of MeOH, EtOH, PrOH, and iso-BuOH on the titration results was also [illegible]. I, II, III, and IV can be used as indicators for titration of strong acids by strong bases. In the presence of alcs, the titration end point appeared at lower pH values. Good results were obtained with II and IV by adding iso-BuOH. Titration of weak acids by strong bases with I, II, III, and IV gave good results in the absence of alcs. Titration of strong bases by weak acid was possible only in presence of iso-BuOH.

Z. Kurtyka

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MICHALSKI, EUGENIUSZ

6

Electrometric determination of silver in ammonium hydroxide solution by depolarization of the electrode. Eugenius Michalski and Włodzimierz Jedrzejewski (Univ. Łódź, Poland). Roczniki Chem. 29, 802-70(1955)(English summary).—Electrometric dets. of Ag in NH_4OH was carried out in the presence of excess Cu^{++}, Pb^{++}, and Fe^{+++} by titration with KI soln. The accompanying elements were kept in soln. in the form of tartrate complexes. The end point was established either graphically or from galvanometer deviations in a closed circuit. In ten-fold excess of Fe and twenty fold excess of Cu and Pb the error was less than 0.5%.

P. Dreyfuss

PM

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MICHALSKI, E.

MICHALSKI, E. Definition by the coulometric method in chemical analysis. p. 492.

Vol. 9, no. 10, Oct. 1955
WIADOMOSCI CHEMICZNE
SCIENCE
Poland

So: East European Accession, Vol. 6, No. 5, May 1957

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

APPROVED FOR RELEASE: 06/23/11: CIA-RDP86-00513R001033800024-6

Abs Jour : Referat Zhur - Khimiya, No 3, 1957, 8459

surface of ~0.3 cm^2 (in the first case, the reference electrode is dipped into a 0.02 N KI solution to which 1% alcoholic I_2 solution is added with constant stirring, and in the second case, into a saturated TlI solution). In the first case the error in the determination of solutions of concentration 0.1-0.05 N is from -0.5 to -1.2%, and in the second case, from 0.3 to 0.6%. When more dilute solutions are used in the titration greater errors are obtained. The presence of secondary electrolytes affects the results from the determinations. The advantages of the method consist in the simplicity of the equipment in comparison to the potentiometric method used up to the present time.

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Michalski, E.

Poland/ Analytical Chemistry - Analysis of Inorganic Substances G-2

Abs Jour : Referat Zhur - Khimiya, No 3, 1957, 8459

Author : Michalski, E. and Jedrzejewski, W.
Inst : Lodz University
Title : The Electrometric Determination of Salts of Univalent Thallium by the Polarization of the Indicator Electrode

Orig Pub : Zesz. nauk. Univ. Lodzkiego, ser. 2, 1955, No 1, 127-131 (in Polish with summaries in English and Russian)

Abstract : The determination of salts of Tl^+ have been carried out by titrating a known amount of KI with the solution to be analyzed. The end point of the titration was determined by the return of the galvanometer needle to the zero position, caused by the polarization of the indicator electrode (IE) at the equivalence point. The system consists of 2 half-cells, the IE and the reference electrode, connected by a salt bridge filled with a saturated KNO_3 solution and of a mirror galvanometer with a sensitivity of $1.9 \pm 0.5 \times 10^{-8}$ amps/division. The IE is prepared from Pt-wire having a diameter of 0.5 mm and a length of 4 mm; the reference electrode may be prepared either from Pt wire or from a Pt sheet with a total

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polarization of the indicator electrode (IE) in consequence of the elimination of the depolarizing ions I^-. The system consisted of two half-cells: IE and the comparison electrode (CE) connected with an electrolytic switch with a saturated solution of KNO_3, and of an galvanometer, sensitivity 1.9×10^{-8} a. IE and CE were made of Pt wire 0.5 mm in diameter and about 3 mm long. IE was submerged in the solution of KI, and CE was submerged in the solution of $Hg_2(NO_3)_2$, the concentration of which did not influence the determination result. The above system does not need any additional current source. The average errors of determination were: at 0.1 n. solution of

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MICHALSKI, EUGENIUSZ

POLAND/Analysis of Inorganic Substances G-2

Abs Jour: Ref Zhur-Khimiya, No 6, 1957, 19553

Author : Eugeniusz Michalski, H. Filipczuk
Inst : Lodz University
Title : Electrometric Determination of Salts of Monovalent Mercury by Method of Polarizing One of Electrodes.

Orig Pub: Zesz. Nauk. Univ. Lodzkiego. Nauki Matem. Przyrodn., 1955, No 1, 135 - 143.

Abstract: The salts of $Hg_2(1+)$ are determined by the method of reversed titration of a known quantity of KI by the analysed solution of the $Hg_2(1+)$ salt. The titration end is estimated by the disappearance of the current in the circuit caused by the

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MICHALSKI, Eugeniusz (Warszawa, ul. Bialobrzeska 17 m 33)

Visceral lupus erythematosus. Polski tygod. lek. 9 no.42:1362-1365 18 Oct 54.

1. Z Oddzialu Chorob Wewnetrznych I.D.S.K.L.; kierownik: doc. W.Hartwig, w Instytucie Gruzlicy; dyrektor: prof. J.Misiewicz.
(LUPUS ERYTHEMATOSUS,
visceral)

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Michalski, E.

V 407. Electrometric determination of iodides based on the polarisation of the electrode. E. Michalski and M. Zuk-Kunnazewska (*Łódz Towarz. Nauk, Section III*, 1954, No. 34, 10 pp.).—A solution of an iodide to be titrated is connected by a bridge of KNO_3 with a satd. $HgNO_3$ soln. Immersed in each soln. are platinum electrodes, connected through a galvanometer. A small current is registered which is due to the reaction $2I' \rightleftharpoons I_2 + 2e$. As the iodide soln. is titrated with $AgNO_3$ the iodide concn. decreases because of the formation of AgI, and the current drops. At the equivalence point a sudden fall in the concn. of iodide produces polarisation, and the current stops. Iodide solutions of 0·004 to 0·1 *N* can be determined with a mean error of 0·05 per cent. Errors of 0·2 and 1 per cent., respectively, are obtained in the presence of chlorides and bromides. CHEM. ABSTR.

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