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351

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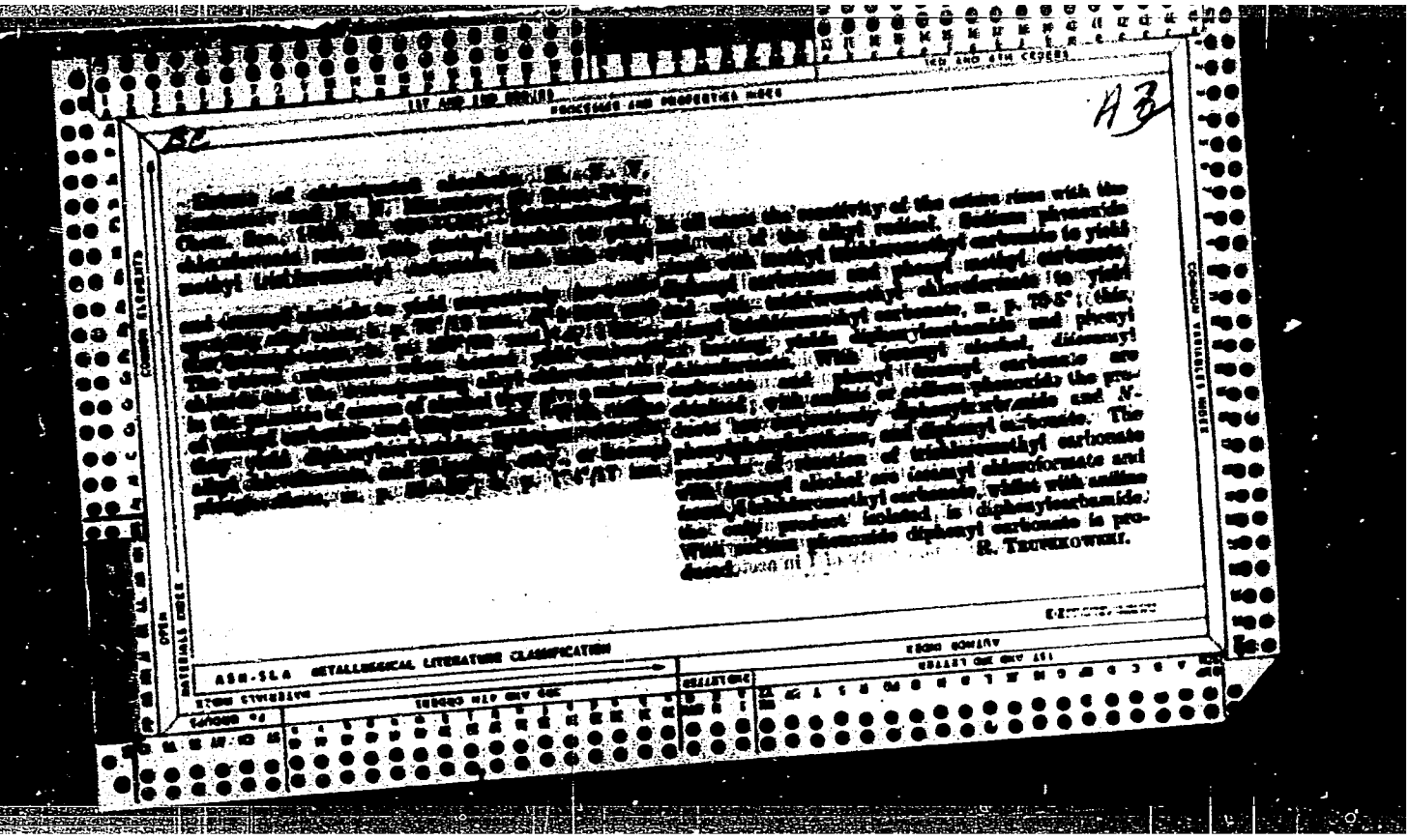

R. TRUMKOWSKI.

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

10

Reaction of complex esters containing a trichloromethoxyl group. IV. Action of aluminum chloride on alkyl trichloromethyl carbonates. N. N. MELNIKOV. *J. Russ. Phys.-Chem. Soc.* **62**, 2019-22 (1930), cf. *C. A.* **24**, 5725. The action of $AlCl_3$ on Me, Et and Pr trichloromethyl carbonates gives CO_2, $COCl_2$ and the corresponding alkyl chloride.

B. C. A.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

MATERIALS INDEX

AUTHOR INDEX

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

10

Ca

Action of anhydrous sodium acetate on alkyl trichloromethyl carbonates. N. N. Melnikov and E. A. Gnomova. *J. Gen. Chem.* (U. S. S. R.) 1, 740-2(1931).—The action of compds. of the phosgene group on salts and acids is discussed. The action of anhyd. NaOAc on Ph, o- and p-tolyl and iso-amyl trichloromethyl carbonates is described. Ac_2O and alkyl acetate are obtained. The mechanism of the reaction is not clear. M. and S. suppose that at first the decompn. of the trichloromethyl carbonate decomps. into phosgene and chloroformate which then react with the AcONa.
E. Gurewitsch

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Oxidizing properties of halonitromethanes. A. E. KRUTOV AND N. M. MELNIKOV. *J. Gen. Chem.* (U. S. S. R.) 2, 202–7(1932).—Nitro compds. act as strong oxidizing agents, undergoing some intramol. oxidation with disintegration of the mol. (Nef, *Ann* 280, 273(1894)), while halonitro derivs. react with even greater oxidizing energy, undergoing highly complex reactions and producing a considerable no. of decompn. products. The oxidizing properties of chloropicrin (I) and bromopicrin (II) were further established (cf. Ray, Guha and Das, *C. A.* 14, 1208; 16, 1953; Nekrasov and M., *C. A.* 24, 87) by the pptn. of I in the interaction of I and KI (Sulchev, *C. A.* 25, 4219) by their reactions with KI and alkali sulfides and hydrosulfides and mercaptans. I *and NaHS* Pure cryst. $NaHS.2H_2O$ (27 g.) was dissolved in 250–300 cc. of EtOH by refluxing, the air being expelled from the system by the alc. vapors; then 15.3 g. of I was introduced drop by drop in about 1.5 hrs. while the soln. was kept boiling. In some expts. the escaping gases were absorbed in satd. alc. KOH, while for the detn. of such gases as N_2O and CO_2 in the mixts. sep. expts. were required. Besides CO_2 there was obtained 410–40 cc. gases contg. NO 14.6–17.2, CO 2–4.4, O 0.6–1, and N 79.2–80%. The reaction flask contained 3.9–4.1 g. of elementary S, 2.4–5.5 g. of NaCl and 0.3–0.9 g. of $NaNO_3$. NH_3 and NH_4OH were detected but not detd. CS_2 absorbed in alc. KOH and converted quantitatively to CS(OEt)SK was pptd. as the Cu salt and analyzed by titrating with I and detg. the S. I *and* K_2S. Under similar conditions, 22.4 g. of pure cryst. $K_2S.2H_2O$ in 250 cc. of alc. produced with I, besides COS, 510–40 cc. of gases contg. NO 22.54–25, COS 30–33.7, O 0.7, CO 4–6 and N 33.1–35.7%. No NH_3 and NH_4OH were found. A salt cake (19.8–20 g.) was formed contg. mostly KCl, 0.4 g. of KNO_3 and 0.5 g. S. In the alc. KOH was found considerable EtOCSOK formed by the interaction of COS and alc. KOH. No CS_2 was found in the alc. KOH. II *and* Na_2S. To 60 g. of $Na_2S.9H_2O$ in 250 cc. of alc. was slowly added 75 g. of II in 75 cc. of alc., and then the mixt. was heated 15–30 min. to complete the reaction. The gas contained CO_2 30–5, NO 12–4.

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Trichloromethyl and alkyl aryluretheans. I. N. N. MELNIKOV AND M. M. VINOKUROV. *J. Gen. Chem.* (U. S. S. R.) 2, 484-90(1932); cf. *C. A.* 24, 2720, 5725, 25, 4228; 26, 3234.—$Cl_3COCOCl$ (I) and the compds. contg. the $OCCl_3$ group react with amines in analogy with alcs. and phenols, giving the same end products as in the reaction with $COCl_2$: I + $8RNH_2 \longrightarrow 2CO(NHR)_2 + 4RNH_2.HCl$. The mechanism of the reaction without formation of intermediate products is not clear and was investigated. To 20 g. I in 150 cc. Et_2O was slowly added with cooling and shaking 27.5 g. p-$C_6H_4(NO_2)NH_2$ (II) in 150 cc. Et_2O, then filtered from the mixt. of II.HCl and sym. $CO(NHC_6H_4NO_2)_2$ (III), the Et_2O distd. off *in vacuo*, dissolved in CCl_4 to remove insol. III, the CCl_4 distd. off *in vacuo*, giving 13 g. of unstable *trichloromethyl p-nitrophenyluretheon*, $Cl_3COCONHC_6H_4NO_2$ (IV): I + 2II $\longrightarrow$ IV + $Ph_2NH.HCl$. By boiling with H_2O or on standing in moist air IV is decompd. into III and p-$C_6H_4(NO_2)NH_2$: IV + $2H_2O \longrightarrow 2CO_2 + 3HCl + C_6H_4(NO_2)NH_2$. IV (14 g.), heated 3 hrs. at 150-60° with 12 g. anhyd. AcONa and then digested with H_2O, gave 6 g. of ppt. of p-$C_6H_4(NO_2)NHAc$, m. 207°. IV *per se* or in CCl_4, heated 4 hrs., gave p-nitrophenylisocyanate, p-$O_2NC_6H_4NCO$, crystals from CCl_4, m. 57°: IV $\longrightarrow COCl_2 + HCl + C_6H_4(NO_2)NCO$. IV produced with alcs. alkyl derivs. and dialkyl carbonates: $CO(OMe)NHC_6H_4NO_2$, m. 180°, and $CO(OMe)_2$ b. 90°, d_4^{20} 1.069, n^{20} 1.3684; $CO(OEt)NHC_6H_4NO_2$, m. 139°, $CO(OEt)_2$, b. 124°, d_4^{20} 1.069, n^{20} 1.3684; $CO(OPr)NHC_6H_4NO_2$, m. 116°, $CO(OPr)_2$, b. 167°, d_4^{20} 0.9414, n^{20} 1.4016; $CO(OBu\text{-}iso)NHC_6H_4NO_2$, m. 81°, $CO(OBu\text{-}iso)_2$, b. 186°, d_4^{20} 0.9130, n^{20} 1.4076; $CO(OAm\text{-}iso)NHC_6H_4NO_2$, m. 98°, $CO(OAm\text{-}iso)_2$, b. 228° (uncor.), d_4^{20} 0.907, n^{20} 1.4173. To 20 g. I in 25 cc. Et_2O was added with cooling and shaking 33.8 g. Ph_2NH in 120 cc. Et_2O, the soln. filtered from the mixt. of $Ph_2NH.HCl$ and $CO(NPh_2)_2$, the Et_2O distd. off and the residue recrystd. from Et_2O, giving *trichloro-*

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

methyl diphenylurethan, $CO(OCCl_3)NPh_2$ (V), m. 61°. V heated 4 hrs. at 200–60° is decompd. into $COCl_2$ and diphenylcarbamyl chloride, $CO(Cl)NPh_2$, crystals from CH_2Cl_2, m. 84°. V boiled 4 hrs. with H_2O gave Ph_2NH, m. 53°. V produced with alks. alkyl diphenylurethans and dialkyl carbonates: $CO(OMe)NPh_2$, m. 85°; $CO(OEt)NPh_2$, m. 84°; $CO(OPr)NPh_2$, m. 85°; $CO(OBu\text{-}iso)NPh_2$, m. 87°; $CO(OAm\text{-}iso)NPh_2$, m. 84°. V heated 2 hrs. at 145–50° with anhyd. AcONa gave Ph_2NAc and Ac_2O.

CHAS. BLANC

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

Ca

10

Phosgene. N. N. Melnikov. *J. Chem. Ind.* (Moscow) 1932, No. 9, 20-5.—
Review with 156 references.
H. M. Leicester

COMMON ELEMENTS

MATERIALS INDEX

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

10

The polymers of phosgene. N. N. Melnikov. *Voennaya Khim.* 1934, No. 2, 9–12; *Chem. Zentr.* 1935, I, 1660.—A review of the properties of diphosgene and other alkyl and chloral trichloromethyl carbonates. W. A. M.

COMMON ELEMENTS

MATERIALS INDEX

ASG-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

10

PROCESSES AND PROPERTIES INDEX

Reactions of carbonic acid derivatives. N. N. Mel'nikov. *J. Gen. Chem.* (U. S. S. R.) 4, 1057 (1954).

The following reactions were studied: (1) ROC(:O)OC-Cl_2 + H_2O, where R is Ph, p-MeC_6H_4, o-MeC_6H_4 and iso-Am. The reaction gave CO_2, ROH, HCl and $COCl_2$. (2) The same derivs. of carbonic acid, except iso-Am, + NaOH, giving NaCl, Na_2CO_3, ROH and H_2O. (3) Me and Et derivs. + NH_3, giving $CO(NH_2)_2$, NH_4Cl and $ROC(:O)NH_2$. (4) Me deriv. + EtMgBr, giving Et_3COH, MeOH, $MgCl_2$ and $MgBr_2$. The mechanism of the reaction with H_2O is explained on the basis of sapon. with the formation of $HOCCl_3$ which decomposes into $COCl_2$ and HCl.

S. L. Madorsky

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

10

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Aliphatic nitro compounds. N. N. Mel'nikov. *J. Gen. Chem.* (U. S. S. R.) 4, 1061-3(1934).—$MeNO_2$, heated with H_2SO_4 at 100-10°, decomposed into CO_2, CO, NH_3 and NH_4OH. $MeNO_2$ vapor, heated to 350-400°, decomposed into H_2O, CO_2, CO, N_2 and NH_3. $CHBr_2NO_2$, heated with H_2SO_4 to 130-5°, gave HBr, CO and $HNOSO_4$. CBr_3NO_2, heated with H_2SO_4 at 100°, gave $COBr_2$ and NOBr. CBr_3NO_2, heated with KOH, gave N_2, CO, NO, K_2CO_3, KBr, $KBrO_3$, KNO_3 and KNO_2.

S. I. Madorsky

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Chloroprene. N. N. Melnikov. *Voennaya Khim.* 1934, No. 5, 7-11; *Chem. Zentr.* 1935, I, 1650. A review.
W. A. Moore

COMMON ELEMENTS

OPEN

MATERIALS INDEX

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

PROCESSES AND PROPERTIES INDEX

A-3

BC

Organic compounds of thallium. S. S. NAMETKIN and N. N. MELNIKOV (Compt. rend. Acad. Sci. U.R.S.S., 1935, 1, [illegible]—234).—The following compounds have been obtained from the corresponding diazonium chlorides and $TiCl_3$: $Ph \cdot N_2 \cdot TiCl_4$ (I), m.p. [illegible]; m-$C_6H_4Me \cdot N_2 \cdot TiCl_4$, m.p. 50°; p-$OH \cdot C_6H_4Me \cdot N_2 \cdot TiCl_4$, m.p. 53°; p-$OEt \cdot C_6H_4 \cdot N_2 \cdot TiCl_4$, m.p. [illegible]°; p-, m.p. 91°, and m-$NO_2 \cdot C_6H_4 \cdot N_2 \cdot TiCl_4$, m.p. 111—112·5°; o-$(NO_2 \cdot C_6H_4 \cdot N_2)_2 \cdot TiCl_4$, m.p. 98—98·5°; α-$C_{10}H_7 \cdot N_2 \cdot TiCl_4$, m.p. 71—72·5°. The NO_2-compounds have three and the other compounds two ionisable Cl atoms. (I) decomposes with the formation of Ph_2, PhCl, TiCl, and (in presence of H_2O) PhOH. A method for the determination of Tl is described, depending on the oxidation of the org. compound by $KMnO_4$ and H_2SO_4 followed by iodometric titration. W. O. K.

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

10

Action of phenylmagnesium bromide on iodoaryl dichlorides. N. N. Mel'nikov. *J. Gen. Chem.* (U. S. S. R.) 5, 28-9(1935). The mechanism of the reaction between an iodoaryl dichloride and RMgBr and RMgI (R = alkyl or aryl) (cf. Hepworth, *C. A.* 19, 3632; Zappi and Degiorgi, *C. A.* 20, 113, 1583) was studied in the interaction of PhMgBr with $PhICl_2$ and p-$MeC_6H_4ICl_2$ (I). In the reaction of PhMgBr with $PhICl_2$ were isolated PhCl, PhBr, PhI, Ph_2, and Ph_2ICl, and in that with I, PhCl, PhBr, MeC_6H_4I, Ph_2, p-MeC_6H_4Ph and $Ph(MeC_6H_4)ICl$. The results differ somewhat from those obtained by H. and Z. and D. The 2 reactions can be represented by the following general scheme: $RICl_2 + 2PhMgBr \rightarrow RIPh_2 + MgCl_2 + MgBr_2$, with the intermediate formation of the unstable R_2I; this on decomp. enters into 2nd reactions with the formation of several products: $RIPh_2 \rightarrow RPh + PhI$; $RIPh_2 + RICl_2 \rightarrow 2RI + 2PhCl$. The formation of Ph_2 and MeC_6H_4Ph is explained as a secondary reaction between the iodoaryl and PhMgBr. The formation of the iodonium compds. can be caused in the decompn. of the org. Mg complex with HCl: $R_2I + HCl \rightarrow RH + R_2ICl$. The formation of PhBr is not clear. It is, probably, formed in the oxidation of PhMgBr by $PhICl_2$. $PhMgBr + PhICl_2 \rightarrow PhBr + PhI + MgCl_2$ (H., *loc. cit.*). The reaction of $PhICl_2$ with BrMgC:CMgBr is highly complicated, giving a considerable no. of compds. of trivalent I, which are difficult to sep. In this reaction there is liberated free C, which had not been observed before in the reactions with org. Mg compds. The reaction proceeds, probably, according to the scheme: $PhICl_2 + BrMgC:CMgBr \rightarrow MgCl_2 + MgBr_2 + Ph$-

Chas. Blanc

METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

BC

A-3

Organic derivatives of thallium. I. Determination of thallium in organic compounds. II. Reaction of thallium chloride with diazo-compounds. S. S. NAMETKIN and N. N. MELNIKOV (J. Gen. Chem. Russ., 1936, 5, 371—372, 373—377).—I. Lehmann and Rupp's method (B., 1911, 508) is applicable to the determination of Tl.

II. The compounds, $R\cdot N_2\cdot TlCl_2$, have been prepared by adding $TlCl_3$ to a $R\cdot N_2Cl$ at −5°: R=Ph, decomp. [illegible]; R=m-C_6H_4Me, decomp. 56°; R=p-C_6H_4Me, decomp. [illegible]; R=p-OEt-C_6H_4, decomp. [illegible]; R=p-$NO_2C_6H_4$, decomp. 91°; R=m-$NO_2C_6H_4$, decomp. [illegible]; R=m-C_6H_4, decomp. [illegible]; [illegible] affords [illegible]. The compounds decompose thus: $R\cdot N_2\cdot TlCl_2$ (I) → $TlCl_2+N_2+RCl$; 2(I) → $2TlCl_2+N_2+R_2+Cl_2$; $TlCl_2$ → $TlCl+Cl_2$; (I)+H_2O → $TlCl_2+R\cdot OH+N_2+HCl$. [illegible] N. T.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

1

Studies in the field of organic analysis. I. Determination of sulfur in certain organic compounds. N. N. Mel'nikov. *J. Gen. Chem.* (U. S. S. R.) 5, [illegible] (1935).—Heat 0.2-0.5 g. of the substance with 10-15 cc. of H_3PO_4 (d. 1.7). To the hot mixt. add gradually, with stirring, 1.5-4 g. of finely powd. $KMnO_4$ (explosions sometimes take place). After 10-15 min. remove excess of $KMnO_4$ with H_2O_2. Dil. the mixt. with 300-400 cc. of water, add HCl and det. the H_2SO_4 with $BaCl_2$ as usual. The method is not suitable for the detn. of S in volatile compds., thiocyanates and heterocyclic S compds. difficultly oxidized. **II. Determination of copper in organic compounds.** N. N. Mel'nikov. *Ibid.* 841-2.—Proceed with the treatment of a sample with H_2SO_4 and $KMnO_4$ as in the detn. of Tl (cf. *C. A.* 29, 81[illegible]). After 15-20 min. add 20-5 cc. of water and remove the excess of $KMnO_4$ with oxalic acid or H_2O_2, boiling some time if the latter is used. Cool, dil. with water to 100-250 cc., add 0.5 g. of KI, 20-5 cc. of 10% KSCN and titrate with $Na_2S_2O_3$ in the presence of starch soln. by Bruhn's method (*C. A.* 12, 2293; 13, 939; 15, 351). The method is accurate to 0.15-2%. Chas. Blanc

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

10

Chemistry of the organic compounds of thallium. III. Synthesis of the organic compounds of thallium with simple substituents in the aromatic radical. S. S. Nametkin, N. N. Mel'nikov and O. P. Gracheva. *J. Gen. Chem.* (U. S. S. R.) 9, 1455-9(1935). cf. C. A. 29, 2220. The synthesis of aromatic Tl compds. substituted in the ring with a halogen or OMe group is based on the following reactions: (1) XArMgBr + $TlCl_3$ → (XAr)$_2$TlBr + $MgCl_2$ (cf. Hein and Spate, C. A. 30, 2668). The reaction proceeds similarly to the synthesis of unsubstituted aryl Tl compds. with 35-50% yields. (2) $ArB(OH)_2$ + TlX_3 + H_2O → $ArTlX_2$ + HX + $B(OH)_3$ and (3) $2ArB(OH)_2$ + TlX_3 + $2H_2O$ → Ar_2TlX + 2HX + $2B(OH)_3$ (X = Cl or Br) (cf. Challenger and Richards, C. A. 29, 4393). $ArTlX_2$ are easily obtained by boiling $ArB(OH)_2$ with a large excess of TlX_3 in H_2O for 20-30 min., and Ar_2TlX with an equimol. amt. of TlX_3 by boiling for 6 hrs. All these compds. are cryst. substances, melting at high temps. with decompn., insol. in most org. solvents and cannot be purified because of decompn. $ArTlX_2$ are bright yellow or orange and Ar_2TlX colorless. Unless otherwise stated the following compds. were prepd. from $ArB(OH)_2$: p-$BrC_6H_4TlCl_2$, m. 262°, p-$BrC_6H_4TlBr_2$, decompg. 190-200°, p-$ClC_6H_4TlCl_2$, m. above 250°, p-$ClC_6H_4Br_2$, does not m. 250°, $PhCH_2TlBr_2$, m. 205°, p-$(BrC_6H_4)_2TlCl$, does not m. 250°, p-$(BrC_6H_4)_2TlBr$, does not m. 250°, prepd. by the methods 1 and 3. p-BrC_6H_4MgBr (from 0 g. Mg and 0.25 g./mol. p-$C_6H_4Br_2$) was treated with 0.05 g./mol. of $TlCl_3$ in Et_2O in the cold, the mixt. was refluxed on the water bath for 30-40 min., the complex was decompd. with HBr, the ppt. was filtered off and washed with H_2O, alc. and Et_2O. It was freed from impurities by extn. with alc. and Et_2O in a Soxhlet app. and crystd. from C_6H_5N. $(C_6H_5OMe)_2TlBr$, does not m. 250°, was obtained as above by starting from p-BrC_6H_4OMe. It was purified by extn. with hot C_6H_5N and ppta. with H_2O. $(ClC_6H_4)_2TlCl$, $(ClC_6H_4)_2TlBr$, decompg. 240°, was prepd. by the methods 3 and 1 (starting with p-ClC_6H_4Br) as above. Chas. Blanc

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

CA

10

New method of production of organic dihalothallium compounds. N. N. Mel'nikov and G. P. Gracheva. *J. Gen. Chem.* (U. S. S. R.) 5, 1186-90(1935); cf. *C. A.* 29, 81[2]. —Tl compds. of the type $RTlX_2$ (R = alkyl or aryl, X = Cl or Br) are obtained by heating TlX_3 with R_2TlX in water and filtering off the sepd. $RTlX_2$. The yields are affected by the side reaction: $R_2TlX + TlX_3 \rightarrow Tl_2X_3 + 2RX$. $RTlX_2$ are easily decompd. by the action of iodides or on heating in water: $RTlX_2 \rightarrow RX + TlX$; $2RTlX_2 \rightarrow R_2TlX + TlX_3$. R_2Tl also reacts with TlX_3: $2R_2Tl + TlX_3 \rightarrow 3R_2TlX$. The violent, exothermic reaction is accompanied by the side reactions: $R_2Tl + TlX_3 \rightarrow R_2TlX + RX + TlX$; $2R_2Tl + TlX_3 \rightarrow 2R_2TlX + TlX + R_2$. Compds. prepd. were: $MeTlBr_2$, decompg. 140-60°; $EtTlCl_2$, decompg. 180°; $EtTlBr_2$, decompg. 140-60° (deriv. $EtTlBr_2$-NC_5H_5); iso-$AmTlCl_2$, decompg. 210°; iso-$AmTlBr_2$, decompg. 95-110°; $PhTlBr_2$, m. 151-2°; p-$MeC_6H_4TlCl_2$, m. 221-3°; p-$BrC_6H_4TlCl_2$, m. 263°; p-$ClC_6H_4TlCl_2$; $PhTlCl_2$, decompg. about 240°.

Chas. Blanc

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

PROCESSES AND PROPERTIES INDEX

Selenious anhydride as an oxidizing agent in organic chemistry. N. N. Mel'nikov. *Uspekhi Khim.* 5, 443-50 (1936). SeO_2 is a sp. agent for oxidation to the aldehyde or ketone stage with only small amts. of acid formation. SeO_2 can be satisfactorily used as an oxidizing agent for oxidation of paraffins, olefins and alcs. to glycolaldehydes, of substituted acetylenes to hydroxyacetylenes, of terpenes to terpene ketones, of aldehydes and ketones to keto aldehydes. Cyclic monoketones give 1,2-diketones. Mercaptans give disulfides, sulfides give sulfoxides and sulfones. M. and Vol'kov (unpublished work) show that disulfides on vigorous treatment also yield sulfoxides and sulfones. Amines, alcs. and mercaptans at low temp. also give complex Se derivs., while C-methylamides give amide aldehydes, —N:CMe → —N:C(CHO)—.
F. H. Rathmann

ASM SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Ca

10

Reaction of thallium chloride with some organic lithium compounds. N. N. Mel'nikov and G. P. Gracheva. *J. Gen. Chem.* (U. S. S. R.) 6, 634-5(1936); cf. *C. A.* 30, 3403[9].—R_3Tl results from $TlCl_3$ and RLi with the intermediate formation of R_2TlCl. The reaction is accompanied by a partial reduction of $TlCl_3$ to TlCl and Tl. Li (2.8 g.) and 22 g. EtBr in 150 cc. petr. ether give about 40% EtLi. Addn. of 6.5 g. $TlCl_3$ to EtLi gives Et_3TlCl. PhLi, prepd. in 60% yield from 1.4 g. Li and 16 g. PhBr in abs. Et_2O, treated with 5 g. $TlCl_3$ in Et_2O gives 4.5 g. Ph_3TlCl. p-MeC_6H_4Li, obtained from 1.4 g. Li and 17.5 g. p-MeC_6H_4Br as above, on addn. of 5 g. $TlCl_3$ in abs. Et_2O gives 3 g. $(p\text{-}MeC_6H_4)_3TlCl$. It is probable that in the production of R_3Tl from TlCl and RLi (Birch, *C. A.* 28, 6423[4]), the reaction also proceeds in 2 stages: $2RLi + 3TlCl \rightarrow R_2TlCl + 2LiCl + 2Tl$; $R_2TlCl + RLi \rightarrow R_3Tl + LiCl$. Chas. Blanc

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

Investigation of organic boron compounds. I. Action of halogens and hydrogen peroxide on diarylboric acids. N. N. Mel'nikov. *J. Gen. Chem.* (U. S. S. R.) 6, 636-9 (1936). The synthesis of $RB(OH)_2$ (R = aryl) by various methods is accompanied by formation of about 50% by-products, consisting chiefly of R_2BOH. The conversion of R_2BOH with halogens (Br or Cl) or H_2O_2 into $RB(OH)_2$ is based on the reactions: $R_2BOH + X_2 + H_2O \rightarrow RB(OH)_2 + HX + RX$; $R_2BOH + H_2O_2 \rightarrow RB(OH)_2 + ROH$. In the presence of excess halogen or H_2O_2 the $RB(OH)_2$ are decompd. into $B(OH)_3$ and RX or ROH, resp.: $RB(OH)_2 + X_2 + H_2O \rightarrow RX + HX + B(OH)_3$; $RB(OH)_2 + H_2O_2 \rightarrow PhOH + B(OH)_3$. The R_2BOH were prepd. from the corresponding Mg compds. and iso-Bu borate by the method of König and Scharrnbeck (*C. A.* 25, 927). Ph_2BOH (18 g.) in 200 cc. H_2O is treated, with stirring and cooling with water, with 16 g. Br in 300 cc. H_2O. The filtrate from PhBr (14 g.) is evapd. to dryness at 60°, giving 11 g. $PhBr(OH)_2$, m. 210°. With 32 g. Br resulted 30 g. PhBr and 5 g. $B(OH)_3$. From 21 g. $(p\text{-}MeC_6H_4)_2BOH$ and 16 g. Br resulted 4 g. $p\text{-}MeC_6H_4B(OH)_2$, m. 226-8°, and 14 g. $p\text{-}MeC_6H_4Br$. $(p\text{-}ClC_6H_4)_2BOH$ (12.5 g.) with 8 g. Br gave 5 g. $p\text{-}ClC_6H_4B(OH)_2$, m. 260°, and 8 g. ClC_6H_4Br, b. 195°. $(p\text{-}BrC_6H_4)_2BOH$ (17 g.) with 8 g. Br gave 5 g. $p\text{-}BrC_6H_4B(OH)_2$, m. 264°, and 9 g. $C_6H_4Br_2$, m. 86-7°. $p\text{-}MeOC_6H_4B(OH)_2$, m. 205-7°, (6 g.) and 7 g. $p\text{-}MeOC_6H_4Br$, b. 220°, resulted from 12.2 g. $(p\text{-}MeOC_6H_4)_2BOH$ and 8 g. Br. Ph_2BOH (18 g.) in 300 cc. H_2O is stirred with cooling with 350 cc. of 1% Cl in H_2O, the reaction mixt. is freed from PhCl (5 g.) and the filtrate is evapd. at 60°, giving 3 g. $PhB(OH)_2$. $(p\text{-}MeC_6H_4)_2BOH$ (21 g.) treated with 350 cc. 1% Cl as above gave 12 g. $p\text{-}MeC_6H_4B(OH)_2$, m. 220°, and 7 g. $p\text{-}MeC_6H_4Cl$, b. 161°. $(p\text{-}ClC_6H_4)_2BOH$ (12.5 g.) with 175 cc. of 1% Cl soln. gave 5 g. $p\text{-}ClC_6H_4B(OH)_2$ and 3 g. $C_6H_4Cl_2$, b. 183°. $(p\text{-}BrC_6H_4)_2BOH$ (17 g.) with 175 cc. 1% Cl soln. gave 5 g. $p\text{-}BrC_6H_4B(OH)_2$ and 6 g. ClC_6H_4Br. Ph_2BOH (18 g.) in 150 cc. H_2O is treated dropwise with 11 g. 30% H_2O_2, the PhOH (6 g.) is distd. off with steam, and the residue is evapd. at 60°, giving 7 g. $PhB(OH)_2$. With 9 g. Ph_2BOH and 11 g. H_2O_2 in 100 cc. H_2O resulted 5 g. PhOH and 2 g. $B(OH)_3$. Chas. Blanc

10

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

10

Electrochemical synthesis of organic compounds. N. N. Mel'nikov. *Uspekhi Khim.* 6, 4 41 (1937).—A review, with 211 references. F. H. Rathmann

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

10

ca

Aliphatic nitro compounds. IV. The reactions of halogenated nitromethanes with some organometallic compounds. N. N. Mel'nikov. *J. Gen. Chem.* (U. S. S. R.) 7, 456-63(1937); cf. C. A. 29, 3976[6].—CX_3NO_2 and PhMgBr react to form PhOH, Ph_2 and tarry products. With Et_2Hg the chief reaction is to form EtHgX and Et_3CNO_2, though decompn. to CO, CO_2, NO, N_2, butane and ethylene also occurs. The reaction is slow with Et_2Hg and still slower with Ph_2Hg. CX_3NO_2 and Ph_3P form Ph_3PX_2 (or Ph_3PO if the solvent is EtOH), as well as N_2, NO, CO_2 and CO. These reactions show the oxidizing power of CX_3NO_2. H. M. Leicester

COMMON ELEMENTS

COMMON VARIABLES INDEX

OPEN

MATERIALS INDEX

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

AUTHOR INDEX

1ST AND 2ND LETTER

3RD AND 4TH ORDERS

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

PROCESSES AND PROPERTIES INDEX

Some tetraarylphosphonium chlorides. N. N. Mel'nikov, A. E. Kretov and B. I. Mel'tser. *J. Gen. Chem.* (U. S. S. R.) 7, 461–3(1937).—Halogen-substituted ketones or benzyl derivs. react with Ph_3P to give *triphenylacetonylphosphonium chloride* m. 214° (decompn.), its *bromide* m. 221°, *triphenylphenacylphosphonium bromide* m. 253° (decompn.), *triphenyl-o-nitrobenzylphosphonium chloride* m. 230° (decompn.), its *m-isomer* m. 247° (decompn.), its *p-isomer* m. 247° (decompn.) and *triphenyl o-cyanobenzylphosphonium chloride* m. 244–5°.

The NO_2- and CN-substituted compds. undergo some oxidation during their prepn. H. M. Leicester

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

The reaction of diethyl mercury with some acid chlorides. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 7, 464-6(1937). Et_2Hg reacts with AcCl, isovaleryl chloride, BzCl, MeOCOCl and EtOCOCl to give EtHgCl and the corresponding ketone. The ketone is hard to sep. from the EtHgCl and the yields are therefore very small. The reaction is practical only for synthesizing ketones of high mol. wt., or for detg. the mobilities of the halogen or hydrocarbon radicals involved. H. M. Leicester

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

PROCESSES AND PROPERTIES INDEX

10

The reaction of thallium chloride and bromide with some phenols. N. N. Mel'nikov and G. P. Gracheva. J. Gen. Chem. (U. S. S. R.) 7, 467-9(1937). $TlCl_3$ and $TlBr_3$ react with α-naphthol to form $(\alpha\text{-}C_{10}H_7O)_3Tl$, m 74-8°, and with the β-isomer to give $(\beta\text{-}C_{10}H_7O)_3Tl$, m 109-12°. Phloroglucinol adds to $TlBr_3$ to form the highly toxic $C_6H_3(OH)_3.TlBr_3$. Pyrogallol, pyrocatechol and hydroquinone reduce TlX_3 to TlX. Hydroquinone is oxidized to quinone, the others to tars. H. M. L.

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

12

Organic thallium compounds. VII. The synthesis of organic thallium compounds with simple substituents in the aromatic radicals. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 7, 1472-7 (1937).—*m*-MeC_6H_4MgBr and $TlCl_3$ form *di-m-tolylthallium chloride*, m. 245°. The *bromide*, obtained in an analogous way, m. 242°. $(AcOC_6H_4)_3TlBr$ can be prepd from $TlBr_3$ and $(AcOC_6H_4)_2Hg$, but attempts to sapon. it give only tars. However, *p*-borobenzoic acid and $TlBr_3$ give $(HO_2CC_6H_4)_3TlBr$, m. 260°. *Biphenylboric acid*, m. 185-90°, from iso-Bu borate and PhC_6H_4MgBr, reacts with $TlCl_3$ to give *dibiphenylthallium chloride* m. 240-5° (decompn.). The *bromide* (I) does not m. 305°. An analogous reaction gives $(NO_2C_6H_4)_2TlCl$ (II), m. 245° (decompn.). I reacts with $TlBr_3$ to give impure $PhC_6H_4TlBr_2$ m. 185° (decompn.), and II with $TlCl_3$ forms $NO_2C_6H_4TlCl_2$, m. 217° (decompn.). The analogous *dibromide* m. 178° (decompn.). Attempts to nitrate Ph_2TlCl give $PhNO_2$ and $PhTl(NO_3)_2$. H. M. Leicester

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

The mechanism of the oxidation of organic compounds by selenium dioxide. I. The oxidation of alcohols. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 7, 1532-8(1937). - SeO_2 reacts with alcs. to form dialkyl selenites. In this way were prepd. the following esters: Me, Et, Pr, Bu, iso-Bu, b_{11} 113-17°, d_4^{20} 1.2413, n_D^{20} 1.4600, iso-Am, b_{10} 130-1°, d_4^{20} 1.1823, n_D^{20} 1.4585. The esters all saponify easily when they stand in air. When heated to 300° in a N stream, they decomp. to the corresponding aldehyde, Se and H_2O. Further oxidation of the aldehyde produces some CO_2 in most cases. This mechanism is general for the oxidation of alcs. by SeO_2.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

Aliphatic nitro compounds. V. The oxidizing power of bromonitromethanes and -ethanes. N. N. Mel'nikov *J Gen. Chem.* (U. S. S. R.) 7, 1546-52(1937); cf. *C. A.* 31, 4265[8].—PhSH and thiocresol react with CH_2BrNO_2 to form $RSCH_2NO_2$ which decomps. to R_2S_2, CO, N_2 and H_2O. With $CHBr_2NO_2$ the intermediate compd. is $(RS)_2CHNO_2$ which splits to R_2S_2, CO, N_2, NO and H_2O. The reactions with the corresponding ethane derivs. are analogous. When these compds react with Ph_3P, the intermediate compds. are of the type $Ph_3PBrCH_2NO_2$ and the final products are Ph_3PO and Ph_3PBr_2. The oxidizing power of the NO_2 group is increased with the no. of hydrogens on the same C atom. H. M. Leicester

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Ca

10

Organic mercury compounds. II. A method of analysis for water-soluble organic mercury compounds. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 7, 2383-4(1937).—Salts of the type RHgX can be titrated with KCNS in the presence of Fe ion by the Volhardt method, since RHgCNS is very little sol. The method is accurate to 0.3-0.4%. **III. The reaction of mercury dialkyls with the mercury salts of dibasic acids.** *Ibid.* 2518-22.—Mercurydialkyls react with H_2SO_4 or succinic acid without a solvent to give the following salts: $(EtHg)_2SO_4$ (I) m. 198° (decompn.), $(PrHg)_2SO_4$ m. 195° (decompn.), $(BuHg)_2SO_4$ m. 182° (decompn.), $(iso\text{-}AmHg)_2SO_4$ m. 188° (decompn.), *bis(ethylmercuric) succinate* m. 157-8°, *bis(propylmercuric) succinate* m. 133-4°, *bis(butylmercuric) succinate* m. 123-4°, *bis(isoamylmercuric) succinate* m. 133-4°, *bis(phenylmercuric) succinate* m. 215°, decomps. 217°. With $Ba(OH)_2$ I gives EtHgOH. *Ethylmercuric butyrate* b_{16} 170-1° (slight decompn.), d_4^{20} 2.250, n^{20} 1.5241, and *ethylmercuric valerate* b_{16} 171-4° (slight decompn.), m. 23-4°, d_4^{25} 2.1056, n^{25} 1.5172, can be prepd. in a similar way. **IV. The electrochemical preparation of symmetrical mercury compounds from mixed metallo-organic mercury salts.** *Ibid.* 2506-9.—When such compds. as $(RHg)_2SO_4$ are electrolyzed at 20-80° and c. d. 0.01-0.05 amp. per sq. cm., almost quant. yields of R_2Hg are obtained. H. M. Leicester

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

MELNIKOW, N. N.

"Recherches dans le domaine des composes organo-mercuriels. Communication III". Melnikow, N. N., Rokitzkaja, M. S. (p. 2522)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii). 1937, Volume 7, No. 19.

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

MELNIKOV, N. N.

"Recherches dans le domaine des composes organomercuriels. Communication IV". Melnikov, N. N. et Rokitzkaja, M. S. (p. 2599).

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii). 1937, Volume 7, Nos. 20-21.

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

Cu

The mechanism of the oxidation of organic compounds by selenium dioxide. II. Oxidation of ketones and aldehydes. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 7, 2738-46(in French 2747) (1937); cf. *C. A.* 30, 5180[1]; 31, 850[4]. Oxidation of aldehydes and ketones by SeO_2 gives chiefly aldehydo ketones and α-diketones. Se org. compds. and products of deeper oxidation are also formed in small amts. Since Me_2CO and C_6H_5CHO oxidize rapidly while $P_2CH(OEt)_2$ and triacetonemannitol oxidize only after some hydrolysis has occurred, it is evident that carbonyl compds. oxidize only through their enol forms, which give an ester of H_2SeO_3 and decomp. as shown in the 1st paper of this series. H_2SeO_3 reacts with amylene and cyclohexene by adding to the double bond. The addn. compd. decomps. to give Se org. compds., aldehydes, alcs. and higher hydrocarbons. The products of deeper oxidation of carbonyl compds. probably come from a similar addn. to the double bond of the enol form. Oxidations of Me_2CO, AcEt, AcPr and cyclohexanone by SeO_2 are bimol. reactions and the rate is a measure of the degree of enolization of the CO group in these compds. H. M. L.

—Ge[illegible]

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

10

Alkylmercuric salts of dibasic acids. N. N. Mel'nikov and M. S. Rokitskaya. Russ. 53,563, July 31, 1938. Dialkylmercury is treated in equimol. proportions with a mercuric salt of a dibasic acid at 30-200° in the presence of water in amts. up to 20%.

CHEMICAL ELEMENTS

MATERIALS INDEX

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

10

The mechanism of the oxidation of organic compounds by selenium dioxide. III. Oxidation of organometallic compounds. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U.S.S.R.) 8, 834-8(1938); cf. C. A. 32, 2003[9].—Oxidation of R_4Hg (R = alkyl) by SeO_2 at 50-60° gives chiefly $(RHg)_2SeO_4$ and some R_2Se and R_2SeO. Et_2Se (impure), b. 107-12°; $(EtHg)_2SeO_4$, decomp. 200°; $(PrHg)_2SeO_4$, decomp. 220-30°; $(BuHg)_2SeO_4$, decomp. 240-50°; $(iso\text{-}AmHg)_2SeO_4$, decomp. 172°. Ph_3P reacts with SeO_2 in dry CeH_6 at room temp., forming Ph_3PO, m. 153°, and Ph_3PSe, m. 184.5°. In a similar reaction Ph_3As gives Ph_3AsO, m. 170°, and Ph_3AsSe, and Ph_3Sb gives Ph_3SbO, m. 200°, and Ph_3SbSe. C. B.

Tetramethylplatinum and hexamethyldiplatinum. Henry Gilman and M. Lichtenwalter. *J. Am. Chem. Soc.* 60, 3085-6(1938). Me_4Pt and MeNa give 16% of *tetramethylplatinum*, hexagonal crystals from petr. ether, decomps. but does not melt; HCl gives Me_3PtCl. Me_4Pt and K in C_6H_6 give 60% of *hexamethyldiplatinum*, which is not dissocd. at the f. p. of C_6H_6. I in $EtOH$ gives Me_3PtI. C. J. West

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

The mechanism of the oxidation of organic compounds by selenium dioxide. IV. Kinetics of the oxidation of ketones. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) **8**, 1369-80 (in French, 1381) (1938); cf. *C. A.* **33**, 1267[8].—Addnl. exptl. evidence is given to show that the oxidation of ketones by SeO_2 is a bimol. reaction and the rate is a measure of the degree of enolization of the CO group in these compds. (cf. *C. A.* **32**, 2903[3]). The oxidation of the following types of ketones was studied: RCOMe, RCOR, RCOPh and cyclic ketones. Equivs. of ketones and H_2SeO_3 in 75% AcOH were held in a thermostat at 20° and 50° (±0.1°) for 1-6 hrs., and the filtered Se ppts., after washing with H_2O and Et_2O, were dried at 100° to const. wts. The comparative graphs for the series Me_2CO, Et_2CO, Pr_2CO, Bu_2CO and $(iso\text{-}Pr)_2CO$ and Me_2CO, MeCOEt, MeCOPr, Me hexyl ketone, iso-PrCOMe, pinacolone and MeCOPh show that the rate of oxidation decreases gradually with increasing mol. wt. The decline in the rate becomes very sharply defined for isoketones as compared with corresponding normal ketones (Pr_2CO 0.52×10^{-7}, $(iso\text{-}Pr)_2CO$ 0.22×10^{-7}). The gradual decrease in the oxidation rate of MeCOPr, iso-PrCOMe and pinacolone shows the influence of primary, secondary and tertiary radicals on the degree of enolization of ketones. Aliphatic ketones are more easily oxidized (enolized) than aromatic ketones (MeCOPh 0.75×10^{-7}, Me hexyl ketone 1.2×10^{-7} at 20°). The regularity in the decline of oxidation rate with increasing mol. wt. is shown also by aryl aliphatic ketones from MeCOPh to EtCOPh to PrCOPh. Alicyclic ketones are more rapidly enolized than fatty ketones, the rate increasing from 1,2- to 1,3- to 1,4-methylcyclohexanone (cf. Biquard, *C. A.* **31**, 5682[9]). C. B.

ASM-SLA METALLURGICAL LITERAT

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

CA

7

Investigation of organic boron compounds. II. Rapid determination of boron in arylboric acids. N. N. McKinikov. *J. Gen. Chem.* (U. S. S. R.) 8, 1766-7(1938); cf. C. A. 30, 5571[illegible]. The detn. is based on the decompn. of $RB(OH)_2$ and R_2BOH (R = aryl) with excess Br with quant. liberation of H_3BO_3, according to the previously shown reactions: $RB(OH)_2 + Br_2 + H_2O = RBr + HBr + B(OH)_3$; $R_2BOH + 2Br_2 + 2H_2O = 2RBr + 2HBr + B(OH)_3$. The method gives results accurate to 0.1% B, but cannot be used in the analysis of more stable aliphatic and benzylboric acids. Dissolve 0.05-0.2 g. of $RB(OH)_2$ in 50-150 ml. H_2O, cool, add excess of 0.1 N soln. of Br in KBr and allow to stand 2 hrs. Introduce 5-10 ml. of 5% KI and 3 ml. of 5% HCl and titrate the sepd. I with 0.1 N $Na_2S_2O_3$ and starch soln. To det. B in diarylboric acids insol. in H_2O, dissolve a sample in a little 0.5 N NaOH, neutralize carefully with dil. HCl and proceed as above. Chas. Blanc

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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Organic boron compounds. III. Synthesis of aryl- and diarylboric acids. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) **8**, 1768-75(1938); cf. *C. A.* **33**, 4908[7].—The method of König and Scharrnbeck (*C. A.* **25**, 927) is used again in the prepn. of aryl- and diarylboric acids from the corresponding Mg compds. and iso-Bu borate in Et_2O. All these compds. are cryst. white substances, fairly sol. in org. solvents and sparingly sol. in H_2O. When stored over dehydrating agents, these acids form anhydrides. *2,5-$MeClC_6H_3B(OH)_2$* (I) (from 4,2-$ClBrC_6H_3Me$), m. 184-6°. *4,2-$MeBrC_6H_3B(OH)_2$* (from 4,4-$Br_2C_6H_3Me$), m. 157°. *p-$EtC_6H_4B(OH)_2$* (from *p*-EtC_6H_4Br), m. 108-11°. *p-$PhCH{:}CHB(OH)_2$* (from *ω*-bromostyrene), m. 138-41°. *Biphenylboric acid* (from bromobiphenyl), m. 185-90°; as a by-product there is formed a little of *diphenylboric acid*, does not m. 300°. *2,4,5-$Me_3BrC_6H_2B(OH)_2$* (from 4,6,1,3-$Br_2C_6H_2Me_2$), m. 200-11°. *2,4,5-$Me_3ClC_6H_2B(OH)_2$* (from 4,6,1,3-$BrCl-C_6H_2Me_2$), m. 155-7°. *4,5-$MeClC_6H_3B(OH)_2$* (from 2,4-$ClBrC_6H_3Me$), m. 242-7°. *(p-$ClC_6H_4)_2BOH$*, m. 75°, formed as a by-product in the prepn. of *p*-$ClC_6H_4B(OH)_2$ (*loc. cit.*). *(2,5-$MeClC_6H_3)_2BOH$*, m. 81°, is obtained as a by-product in the prepn. of I. *p-$ClC_6H_4CH_2B(OH)_2$* (from *p*-$ClC_6H_4CH_2Br$), m. 140°. The arylboric acids react with $TlCl_3$ and $TlBr_3$ to give Ar_2TlX, which by interaction with Tl halides in H_2O form $ArTlX_2$ (X = Cl or Br) (cf. *C. A.* **30**, 2182[2]). The following new compds. were prepd. *(2,4,5-$Me_3BrC_6H_2)_2TlCl$*, decomp. 208°. *(2,4,5-$Me_3ClC_6H_2)_2TlCl$*, decomp. 248°. *(4,5-$MeClC_6H_3)_2TlCl$*, decomp. 200°. *(2,5-$MeClC_6H_3)_2TlCl$*, m. 238°. *(4,2-$MeBrC_6H_3)_2TlCl$*, decomp. 225°. *(p-$EtC_6H_4)_2TlCl$*, decomp. 200°. *(m-$Me_2C_6H_3)_2TlBr$*, m. 196°. *(2,4,5-$Me_3BrC_6H_2)_2TlBr$*, decomp. 220°. *(2,4,5-$Me_3ClC_6H_2)_2TlBr$*, m. 190-5°. *(4,5-$MeClC_6H_3)_2TlBr$*, decomp. 200°. *(4,2-$MeBrC_6H_3)_2TlBr$*, m. 254°. *(2,5-$MeClC_6H_3)_2TlBr$*, m. 200°. *(p-$EtC_6H_4)_2TlBr$*, decomp. 280°. The above compds. were obtained in 60-95% yields, forming white cryst. substances. *$MeBrC_6H_3TlCl_2$*, m. 174-7°, *$EtC_6H_4TlCl_2$*, decomp. 155°, and *α-$C_{10}H_7TlCl_2$*, m. 144°, are white cryst. compds. *2,4,5-$Me_3BrC_6H_2TlBr_2$*, m. 192°, and *2,4,5-$Me_3ClC_6H_2TlBr_2$*, m. 185-90°, are yellow cryst. compds. The following compds. form orange crystals. *4,5-$MeClC_6H_3TlBr_2$*, m. 185-8°. *$MeClC_6H_3TlBr_2$*, m. 182°. *$MeBrC_6H_3TlBr_2$*, m. 180°. *p-$EtC_6H_4TlBr_2$*, m. 170°. *$Me_2C_6H_3TlBr_2$*, m. 215°. *α-$C_{10}H_7TlBr_2$*, m. 185°. The above compds. were formed in 30-84% yields.

Chas. Blanc

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

10

Ca

Alkylmercury phosphate. N. N. Mel'nikov and M. S. Rokitskaya. Russ. 54,382, Jan. 31, 1939. Mercury dialkyl is heated with an equimol. amount of mercury phosphate to 50–250° in the presence of 20% H_2O.

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

BC

B-II-1

Synthesis of simpler hydroxyalkyldiphenyl compounds. N. N. Melnikov and L. G. Volfson (Proms. Org. Chim., 1939, 6, 637—63[illegible]).—o-C_6H_5Ph-OH is heated with alkyl halides in presence of $ZnCl_2$ to yield 2-hydroxy-3- and -5-mono- and -3:5-di-alkyldiphenyls. R. T.

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

PROCESSES AND PROPERTIES INDEX

2

CA

Mechanism of oxidation of organic compounds by means of selenious anhydride. V. Kinetics in the oxidation of aldehydes. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 9, 1156-61(1939); cf. *C. A.* 33, 4194[8].—The oxidation of CH_3CHO, C_2H_5CHO, C_3H_7CHO, $C_4H_{11}CHO$, $(CH_3)_2CHCHO$ and $(CH_3)_2CH$-CH_2CHO by means of H_2SeO_3 in 75% AcOH was studied and the velocity of this reaction detd. The results show that the oxidation velocity decreases with increasing mol. wt. with the exception of CH_3CHO where the reaction const. is considerably smaller than that of C_2H_5CHO. This is explained by the easy polymerization of CH_3CHO in acid medium. The reaction const. is decreased also in the iso compds. as compared with the normal compds. Thus the reaction const. of C_4H_9CHO is 7.90×10^{-6}, and of $(CH_3)_2CHCHO$ 1.22×10^{-6}. G. Berend

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

CA

The mechanism of oxidation of organic compounds by selenious anhydride. VI. Oxidation of ketones in alcohol solutions. N. N. McUnikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 9, 1808-12(1939); cf. C. A. 34, 1233[illegible]. The oxidation of Me_2CO, $MeEtCO$ and $MePrCO$ was studied in abs. and aq. MeOH, EtOH, BuOH, iso-BuOH and iso-AmOH. The relation of rate consts. to the structure of the ketones is the same as for the reactions in 75% HOAc. The consts. are lower in the abs. alcs. than in HOAc due to the formation of complexes between the alcs. and the ketones. H_2O breaks up these complexes and so raises the consts. The consts. increase with the mol. wt. of the normal alcs., and are highest in iso-BuOH soln. H. M. Leicester

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

MELNIKOV4N8N8

600

1. MEL'NIKOV, N. N., SKLYARENKO, S.I., CHERKASOVA, Ye. M

2. USSR (600)

"The Question of the Electrochemical Rhodanizing of Organic Compounds", Zhur. Obshch. Khim. 9, No. 19, 1939. Institute of Chemical Pharmaceutic (Ionkoy) Technology, Moscow. Received 11 May 1939.

9. [redacted] Report U-1626, 11 Jan 1952.

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Mercury diethide. N. N. Melnikov and M. S. Rokitskaya. *J. Applied Chem.* (U. S. S. R.) 12, 1803-5(in French, 1805)(1939). Et_2Hg was obtained by the Grignard method by using 120 g. Mg and 530 g. EtBr in 2000 ml. abs. ether, followed by removal of the excess Mg by filtration through glass wool and gradual addn. of 305 g. $HgCl_2$ to the boiling mixt. The Mg org. complex was decompd., after cooling, with 5-7% HCl. The ether layer was sepd. and redistd. on a water bath after drying over $CaCl_2$ and the $HgEt_2$ was distd. in vacuo. A 70-85% yield of Et_2Hg was obtained after heating the reaction mass for 20-48 hrs. The prepn. of Et_2Hg from Na-Hg and EtBr was carried out in a glass and an iron app. equipped with an agitator and a dropping funnel. The reaction was carried out at 0-5°, using a 20% excess of a 0.5% Na-Hg. Among the catalysts investigated (AcOEt, acetone, alc. and others) AcOEt was the best, the presence of water and alc. lowering the yield. Ten references.

A. A. Boehtlingk

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

CA

10

Alkylmercuric hydroxides. N. N. Mel'nikov and M. S. Rokitskaya. Zhur. 57,163, May 31, 1940. Aq. or aq. alc. soln. of $(RHg)_2SO_4$ is treated with an aq. soln. of an equivalent quantity of $Ba(OH)_2$ or $Ca(OH)_2$, the $BaSO_4$ or $CaSO_4$ filtered off, and the RHgOH recovered by evapn.

COMMON ELEMENTS

COMMON VARIABLES INDEX

MATERIALS INDEX

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

13

Insecticide and fungicide. N. N. Mel'nikov, E. A. Pokrovskii and L. T. Vol'fson. Russ. 58,537, Dec. 31, 1940. An aq. emulsion or a soln. of alkylhydroxybiphenyls in an org. solvent is specified.

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

02

13

Organic insectofungicides. N. N. Mel'nikov. *Nauch. Issledovatel. Inst. Udobreniyam i Insektofungisidam Ya. V. Samoilova* (Moscow) 1939-39, 91-3; *Khim. Referat.*

Zhur. 1940, No. 5, 89.—Methods for wood preservation with hydroxybiphenyl are described. Insecticidal and fungicidal mineral-oil emulsions of hydroxybiphenyl, moth-proofing substances (thiourea, phenylthiourea, etc.), contact insectofungicides (dihydroxybiphenyl, esters of thiocarboxylic acids, diethylpropylpyridine) and antiseptics for protecting wood (pentachlorophenol, tetrachlorophenol and their phenolates, dihydroxybiphenyl, tetrachlorophenyl, etc.) also were studied. W. R. H.

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

15

CA

Preparation of mercury fungicide of the "granosan" type. N. N. Mel'nikov and M. S. Rokitskaya. *Org. Chem. Ind.* (U. S. S. R.) 7, 387 (1940). Exptl. details for the prepn. of C_6H_5HgCl according to the scheme $2C_6H_5Br + 2Na + Hg \rightarrow (C_6H_5)_2Hg + 2NaBr$; $(C_6H_5)_2Hg + HgCl_2 \rightarrow 2C_6H_5HgCl$. B. Kamich

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

MEL'NIKOV, N. M.

"The Question of the Electrochemical Rhodanation of Organic Compounds II. The Rhodanation of Aromatic Amines," Zhur. Obshch. Khim., 10, No 15, 1940. Lab. of Org. Chem., Moscow Inst. of Fine Chem. Technology. Received 26 March 1940.

Report U-1610, 3 Jan 1952

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

2

The mechanism of the oxidation of organic compounds by selenious anhydride. VII. The oxidation of substituted acetophenones. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 10, 1439-41 (1940); cf. *C. A.* 34, 3676[6].—Since this oxidation goes through intermediate enolization, the reaction rate measures the ease of enol formation. At 30° in 75% AcOH the order of increasing reaction rate is m-NO_2-C_6H_4COMe, p-BrC_6H_4COMe, p-ClC_6H_4COMe, PhCOMe, p-$MeOC_6H_4COMe$, p-MeC_6H_4COMe, p-IC_6H_4COMe, Ph-CH_2COMe. H. M. Leicester

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

The mechanism of oxidation of organic compounds by selenious anhydride. VIII. The oxidation of isomeric ketones. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 10, 1713-16(1960); cf. *C. A.* 15, 2400f.—Oxidation of the isomeric Me Am and Me hexyl ketones by the previously used method shows that the rate of oxidation, and, hence, the rate of enolization, depends greatly on structure. Ketones contg. an even no. of CH_2 groups between the CO and a secondary radical are less enolized than normal ketones, or those contg. an odd no. of CH_2 groups between CO and a secondary radical. Ketones in which a secondary or tertiary group is attached to CO are still less enolized. H. M. Leicester

Physical properties and chemical constitution. VI. Some saturated and unsaturated cyano esters. David M. Cowan and Arthur I. Vogel. *J. Chem. Soc.* 1960, 1528-31; cf. *C. A.* 54, 2707f.—Unsatd. cyano esters of the general formula $CRR':C(CN)CO_2Et$ were prepd. by the condensation of the appropriate ketone with $CH_2(CN)CO_2Et$ in the presence of anhyd. Na_2SO_4 and a little piperidine at 100°; the following *Et α-cyano-β,β-dialkylacrylates* were obtained: *Me Et* (I); *di-Et*, (II); *Me Pr* (III); *Et Pr* (IV); *di-Pr* (V). The satd. cyano esters $CHRR'-CH(CN)CO_2Et$ were prepd. by the reduction of the corresponding unsatd. esters with Al-Hg in moist Et_2O soln. the following were obtained: *Et α-cyano-β-methylbutyrate* (VI); β-methylvalerate (VII); β-ethylvalerate (VIII); β-methylcaproate (IX); β-ethylcaproate (X) β-propylcaproate (XI). The b. pts., n for C, D, F and G lines (in this order) at 20°, surface tensions (γ), and d at several temps. of the aforementioned esters were detd The surface tensions are stated in terms of γ at 20° followed by the temp. coeff. in parentheses, and the d. values are with respect to H_2O at 4°. The mol. refractivities for the C, D, F and G' lines (not recorded here) and the parachors (*P*) were evaluated. The results are as follows: I, b_5 95.5°; n 1.46410, 1.46771, 1.47685, 1.48446; d_{20} 1.0091, d_{20-4} 0.9990, d_{20-4} 0.9488; γ_{20} 34.43 (0.10); *P* 403.6. II, b_5 96.5°; n 1.46343, 1.46692, 1.47594, 1.48427; d_{20} 0.9931, d_{20-4} 0.9501, d_{20-4} 0.9362; γ_{20} 33.53 (0.098); *P* 440.4. III, b_5 100°; n 1.46471, 1.46829, 1.47740, 1.48484; d_{20} 0.9890, d_{20-4} 0.9323, d_{20-4} 0.9335; γ_{20} 33.21 (0.093); *P* 441.2. IV, b_5 104.5°; n 1.46486, 1.46839 1.47732, 1.48468; d_{20} 0.9708, d_{20-4} 0.9[illegible], d_{20-4} 0.9221; γ_{20} 32.46 (0.099); *P* 477.7. V, b_5 116.5°; n 1.46422, 1.46771, 1.47648, 1.48390; d_{20} 0.9630, d_{20-4} 0.9287, d_{20-4} 0.9115; γ_{20} 31.81 (0.095); *P* 516.6. VI, b_4 78.5°; n 1.42041, 1.42256, 1.42273, 1.43160; d_{20} 0.9710, d_{20-4} 0.9307, d_{20-4} 0.9109; γ_{20} 30.46 (0.091); *P* 377.3. VII, b_4 98°; n 1.42576, 1.42797, 1.43316; 1.43680; d_{20} 0.9653, d_{20-4} 0.9310, d_{20-4} 0.9089; γ_{20} 30.78 (0.089); *P* 414.3. VIII, b_4 99°; n 1.43127, 1.43350, 1.43880, 1.44264; d_{20} 0.9608, d_{20-4} 0.9276, d_{20-4} 0.9084; γ_{20} 30.98 (0.092); *P* 450.4. IX, b_4 101.5°; n 1.42976, 1.43199, 1.43722, 1.44103; d_{20} 0.9534, d_{20-4} 0.9199, d_{20-4} 0.8991; γ_{20} 30.11 (0.087); *P* 451.6. X, b_{3-4} 108°; n 1.43306, 1.43620, 1.44145, 1.44531; d_{20} 0.9504, d_{20-4} 0.9185, d_{20-4} 0.8971; γ_{20} 30.40 (0.088); *P* 488.7. XI, b_4 120.5°; n 1.43778, 1.43993, 1.44569, 1.44904; d_{20} 0.9401, d_{20-4} 0.9089, d_{20-4} 0.8900; γ_{20} 30.06 (0.085); *P* 527.1. The corresponding values for Et cyanoacetate were b_4 85.6°; n 1.41540, 1.41751, 1.42263, 1.42639; d_{20} 1.0648, d_{20-4} 1.0262, d_{20-4} 0.9990, d_{10-4} 0.9682; γ_{20} 36.66 (0.11); *P* 302.4. The calcd. parachors, using Sugden's consts. (*C. A.* 19, 197) and mol. refractivities, based on Eisenlohr's refraction equivs. (*C. A.* 6, 1872) were compared with the exptl. values. For most of the esters the observed *P* are less than those calcd., and the mol. refractivities of the unsatd. esters exhibit an exaltation attributed to the conjugated system >C:C.C:N. The *P* difference per CH_2 group varies from 36.1 to 38.9, and hence is always less than the accepted value of 39 to 40 units. S. G.

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

Ca

20

Wood preservation. N. N. Mel'nikov, Z. E. Bekker and M. S. Rokitskaya. Russ. 59,034, Feb. 28, 1941. Wood is impregnated with solns. or emulsions of alkylhydroxybiphenyls or alkylchlorohydroxybiphenyls.

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

ca

10

Organic compounds of mercury. V. The reaction of dialkylmercury with mercuric salts of tribasic acids. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U. S. S. R.) 11, 602-5(1941); cf. *C. A.* 32, 2884[1].—The authors investigated the possibility of prepg. dialkylmercury salts of tribasic acids by a method which is simpler than those available at present and is more adaptable for manufg. purposes. The general method used was the reaction of R_2Hg with mercuric salts of phosphoric, arsenic and nitric acids. A mixt. of 13 g. Me_2Hg, 15 g. $Hg_3(PO_4)_2$ and 0.5 cc. H_2O was heated under a reflux condenser for 0.5 hr. at 100°, cooled and recrystd. from alc., yielding *tris(methylmercury) phosphate*, decomps. 182°, in 80% yield. A mixt. of 25 g. Et_2Hg, 25.5 g. $Hg_3(PO_4)_2$ and 5 cc. H_2O heated under a reflux at 115° for 15 min., cooled and recrystd. from H_2O, yielded 88% of *tris(ethylmercury) phosphate*, m. 170-80°; hydrate (formed on recrystn. from H_2O), m. about 110°. A mixt. of 15.4 g. Et_2Hg and 17.4 g. $Hg_3(AsO_4)_2$ heated under reflux at 120° for 1 hr., cooled and recrystd. from [illegible] yielded 75% of *tris(ethylmercury) arsenate*, m. 184-[illegible]°. To 11.5 g. $Hg(NO_3)_2$ there was added 8.5 g. Et_2Hg and the mixt. heated under a reflux for 15 min. on a steam bath, cooled and recrystd. from H_2O to yield 80.5% *ethylmercury nitrate*, m. 86-6.5°, which crystd. from H_2O as a *monohydrate*. A mixt. of 14 g. Pr_2Hg, 15 g. $Hg_3(PO_4)_2$ and 0.5 cc. H_2O was heated at 170° for 35 min., cooled and recrystd. from benzene to yield 98% of *tris(propylmercury) phosphate*, m. 96°. A mixt. of 18 g. Bu_2Hg, 15 g. $Hg_3(PO_4)_2$ and 0.5 cc. H_2O was heated for 1.5 hrs. at 170°, cooled and recrystd. from benzene to yield 88% of *tris(butylmercury) phosphate*, m. 75°. A mixt. of 19 g. iso-Am_2Hg and 15 g. $Hg_3(PO_4)_2$ heated 2 hrs. at 180°, gave 62% *tris(isoamylmercury) phosphate*, m. 105° (from benzene).

G. M. Kosolapoff

ASM-SLA METALLURGICAL LITERATURE

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

PROCESSES AND PROPERTIES INDEX

10

Preparation of ethylmercuric phosphate. N. N. Melnikov and M. S. Rokitskaya. *J. Applied Chem.* (U. S. S. R.) 14, 440-6(in French, 448)(1941).—A reactor (enameled or made of cast iron) equipped with an agitator and a reflux condenser is charged with equimol. amts. of $Hg_3(PO_4)_2$ and $HgEt_2$ (followed by the addn. of 10% H_2O (on the amt. of substances taken) and heating is carried out during 40-70 min. to 80-100°. Then 200% of H_2O is added after completion of the reaction and the soln. is filtered while hot. The crystn. is effected by evapn. of the sq. soln. The pure and dry product m. 170-80°. 5 references. A. A. Roehtlingk

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

A-4

BC

[illegible] activity of phenols. E. N. [illegible], A. M. Avetesian, and M. S. [illegible] (Compt. rend. Acad. Sci. U.R.S.S., 1941, [illegible]). Data are given for the phenol and mol. phenol coeffs. of a series of [illegible] phenols, p-substituted phenols, and alkyl- and aryl-chlorophenols, using *Paramecium caudatum* as test organism. An increase in the no. of Cl in the phenol mol. increases its effectiveness against *P. caudatum*. There is a pronounced increase in activity in passing from phenol to tetra- and penta-chlorophenol. The least and most active compounds are 2 : 4 : 6-tri- and tetra-chlorophenol, respectively. Various substituents in the p-position to the hydroxyl group increase the activity of phenol, except the nitro- and sulphonic groups which cause a decrease. A more active compound is usually obtained when the p-H is replaced by a hydrocarbon radical. The activity of alkylphenols is increased by increase in mol. wt. of the alkyl radical. The increase is particularly marked with p-benzylphenol. Alkyl- and aryl-chlorophenols are much more active than the corresponding compounds which contain no halogen. It is concluded that the mechanism of the action of phenolic compounds on *P. caudatum* is similar to that on other bacteria, and that certain bacteria and protozoa have pellicles which are similar in constitution and composition.

J. N. A.

COMMON ELEMENTS

OPEN

MATERIALS INDEX

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

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BIII 6

BC

Derivatives of 6-hydroxydiphenyl. N. N. Melnikov, M. S. Rokitskaia, and Z. K. Bochar (Compt. rend. Acad. Sci. U.R.S.S., 1941, 32, 186—187).—Alkylhydroxydiphenyls (I) are prepared from o-C_6H_5PhOH (II) (1 mol.), AlEOH (1 mol.), and anhyd. $AlCl_3$ (2 mols.) at 180—185°; alkylhydroxydiphenyls (III) are similarly prepared from C_6H_5ClPhOH, or from (I) by Cl_2 and SO_2Cl_2. d_2^{20}, n_D^{20}, and PhOH-coeffs. towards six micro-organisms are given for various isomeric mixtures of (I) and (III) in which R = Et, Pr, Bu, iso-C_5H_{11} and C_6H_{13}. Most of them are many times more potent than PhOH or (II), and they are recommended for the preservation of citrus fruits. S. A. M.

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

A-4

BC

Structure and insecticidal properties of organic compounds. N. N. Melnikov, N. D. Sucharova, and M. L. Fedor (*Compt. rend. Acad. Sci. U.R.S.S.*, 1941, 32, 414—416).—Data are given for the min. lethal doses of a series of esters of thiocyanoacetic and α-thiocyanobutyric acids against *Pediculus vestimenti* and *Cimex lectularius* and their eve. Esters of thiocyano-substituted acids are much more potent insecticides than are those of the corresponding chloro-acids and esters having an unsubstituted group in the alkyl group of the fatty acid. The insecticidal efficiency of the alkyl thiocyanoacetates increases with increase in mol. wt. of the alkyl group. In the case of the α-thiocyanobutyrates the most active is the isoamyl ester. In general an increase in the mol. wt. of the radical of the fatty acid causes a decrease in the efficiency of the esters. The insecticidal activity of alkyl thiocyanoacetates and α-thiocyanobutyrates differs only slightly from that of the corresponding propyl esters. (For new compounds see A., 1944, II, 16.) J. N. A.

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

CA

10

Structure and germicidal properties of organic compounds. Derivatives of hydroxytiphenyl. N. N. Mel'nikov, M. S. Rokitskaya and Z. E. Bekker. *J. Applied Chem.* (U. S. S. R.) 16, No. 9/10, 426-32(1943).—See *C. A.* 37, 877°. Esters of α-substituted acids. N. N. Melnikov, N. D. Sukhareva and M. L. Fedder. *Ibid.*, No. 11/12, 426-32.—See *C. A.* 37, 490°.

G. M. Kosolapoff

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

PROCESSES AND PROPERTIES INDEX

Ca

10

Electrochemical thiocyanation of organic compounds. III. p-Substituted aromatic amines. N. N. Mal'tsev and E. M. Cherkasova. *J. Gen. Chem.* (U. S. S. R.) 14, 113-15(1944)(English summary); cf. *C. A.* 38, 3015[4].—The expts. were conducted in a Cu vessel acting as the cathode, with a rotating graphite anode; the temp. used was −5°; the c. d. on the anode ranged from 0.03-0.05 amp./sq. cm.; no diaphragm was used. p-Toluidine (1.5 g.), 3.2 g. NH_4CNS, 2 cc. concd. HCl and 35 cc. aq. EtOH treated with 2 amp.-hrs. of current, yielded, after dilu. and refluxing for 1.5 hrs., 2-amino-6-methylbenzothiazole, m. 135-6°. Similarly, 1.5 g. anisidine, 3.7 g. NH_4CNS, 4 cc. concd. HCl and 12 cc. EtOH-10 cc. H_2O gave 57% 2-amino-6-methoxybenzothiazole, m. 163°. By using phenetidine, 60% 2-amino-6-ethoxybenzothiazole, m. 155-6°, was obtained. When 2.3 amp.-hrs. of current was passed through 1.98 g. p-$H_2NC_6H_4CO_2Et$, 2.74 g. NH_4CNS, 5 cc. concd. HCl and 20 cc. EtOH-6 cc. H_2O there was obtained Et 4-amino-3-thiocyanobenzoate, which, heated to 150°, gave 90% 2-amino-6-carbethoxybenzothiazole, m. 240-1°. Similarly, the corresponding Me ester, m. 200°, was prepd. from p-$H_2NC_6H_4CO_2Me$.

G. M. Kosolapoff

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

CA

2

Mechanism of oxidation of organic compounds by selenium oxide. IX. Oxidation of acetone in the presence of acid catalysts. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U.S.S.R.) 14, 1054-8 (1944); cf. C.A. 38, 3239.—The rate of oxidation of acetone and acetophenone by H_2SeO_3 was studied in the presence of HOAc and H_2SO_4 by exptl. methods previously described. The values of the reaction const. × 10^6 (for acetone at 25°) were 65 in pure water, and 54, 86.3, 144.3, and 272.1 in water contg. 10%, 25%, 50%, and 75% HOAc, resp. In dil. H_2SO_4 the corresponding values were 71.6, 188.2, 301.0, 513.0, and 630.7 for 0.1 *N*, 0.5 *N*, 1 *N*, 1.5 *N*, and 2 *N*, resp. At 30°, the following values were obtained: with acetone in 75% HOAc, 404.7 and in 75% HOAc contg. 0.5 *N* H_2SO_4, 1014; with acetophenone in 75% HOAc, 224.7 and in 75% HOAc contg. 0.5 *N* H_2SO_4, 358.4. The results are explained by postulating that the acids catalyzed the enolization of the ketones, thus rendering them more easily amenable to oxidation by H_2SeO_3. **X. Oxidation of ketones, $Ph(CH_2)_nCOMe$.** *Ibid.* 1059-62.—The rate of oxidation of a series of 4 ketones of general formula $Ph(CH_2)_nCOMe$ by 4 *N* H_2SeO_3 in 75% HOAc at 20° was detd. The reaction-rate const., in mols./l. min., multiplied by 10^6 was 5.84 for PhCOMe, 27.19 for $PhCH_2COMe$, 6.35 for $Ph(CH_2)_2COMe$, and 7.06 for $Ph(CH_2)_3COMe$. The results are explained by the decreasing tendency toward enolization, which is in agreement with the electronic structure of these compds.

J. W. Perry

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MELNIKOV, N. N.

"On the Mechanism of Oxydation of organic Compounds with Selenium Dioxide. X. Oxydation of Ketones of the Type $C_6H_5(CH_2)nCOCH_3$." Melnikov, N. N. and Rokitzkaya, M. S. (p. 1059)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1944, Volume 14, no. 11-12.

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

Ca

10

Mechanism of oxidation of organic compounds by selenium dioxide. XI. Keto-enol tautomerism of keto acids. N. N. Mel'nikov and M. S. Rokitskaya. *J. Gen. Chem.* (U.S.S.R.) 15, 657-60(1945)(English summary); cf. *C. A.* 38, 3220[1]. The kinetics of the oxidation of pyruvic and levulinic acids by SeO_2 at 20° in 75% AcOH was studied. The results indicate that levulinic acid is least enolized, followed in ascending order by pyruvic and acetoacetic acids. The const. of oxidation rate by SeO_2 is very small for Ac, being considerably smaller than that of Me_2CO, thus indicating a negligible degree of enolization of this compd. The results indicate the correctness of the mechanism of such oxidation as proposed by the writers in their earlier publications (*C.A.* 33, 4194[8]).

G. M. Kosolapoff

COMMON ELEMENTS

CPEB

MATERIALS INDEX

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

COMMON VARIABLES INDEX

AUTHOR INDEX

1ST AND 2ND LETTERS

3RD AND 4TH CODES

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

10

Ca

Structure and germicidal activity of organic compounds. II. Alkyl sulfondichloroamides. N. N. Mel'nikov, N. D. Sukhareva, and F. Va. Kavenoki. *J. Applied Chem.* (U.S.S.R.) **18**, 568–70(1945)(English summary); cf. C.A. **37**, 877°. -A series of $AlkSO_2NH_2$ was converted to the corresponding $AlkSO_2NCl_2$ by chlorination of suspensions or soln. of the amides in water; the products, after drying, were distd. under reduced pressure. The chlorination was conducted with ice cooling. The following compds. were isolated: $MeSO_2NCl_2$, m. 83°, b_7 102° (84.3%); $ClCH_2SO_2NCl_2$, b_6 102°, d_4^{20} 1.7044 (30%); $EtSO_2NCl_2$, b_{10} 108–10°; d_4^{20} 1.5151 (60%); $PrSO_2NCl_2$, b_{11} 110°, d_4^{20} 1.4450; $BuSO_2NCl_2$, b_{10} 129–31°, d_4^{20} 1.3630 (33%); *iso-Am*SO_2NCl_2, decomps. on distn., d_4^{20} 1.284 (30%). The germicidal power of the products against *S. aureus* is higher than that of chloramine-B; the max. activity among the straight-chain products was found with the Pr group, while the iso-Am deriv. gave the most potent prepn. (limiting concn., 1:150,000). G. M. Kosolapoff

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

10

Ca

Electrochemical synthesis of organic compounds. V. Preparation of selenocyanates. N. N. Mel'nikov and E. M. Cherkasova (Inst. Fine Chem. Tech., Moscow). *J. Gen. Chem.* (U.S.S.R.) **16**, 1025-8(1946)(in Russian), cf. C.I. **39**, 934. Electrolysis of salts of HSeCN in the presence of org. substances in the anode compartment leads to formation of org. selenocyanates. A Cu vessel served as a cathode and container simultaneously, a rotating graphite anode was used. The app. was cooled with ice water. $PhNMe_2$ (1.2 g.), 2.9 g. KSeCN, 30 cc. EtOH, and 10 cc. H_2O were electrolyzed at an anode d. 0.007-0.008 amp./sq. cm. for a total of 1.2 amp. hrs., after diln. with water and extn. with Et_2O there was obtained 15% *1-dimethylamino-4-(cyanoseleno)benzene*, m. 105-6° (from petr. ether). If the reaction is run as above, but with addn. of 1.13 g. concd. HCl and the use of 20 cc. EtOH, the yield is 20%. Much Se is formed during the reaction, coating the electrodes, etc. Similar reaction with Et_2NPh at 0.03-0.008 amp./sq. cm. gave 20% of the *di-Et deriv.*, m. 50-2° (from petr. ether). $PhNHMe$ (3.5 g.), 14 g. KSeCN, 36 cc. EtOH, and 40 cc. water gave, at 0.01-0.02 amp./sq. cm., about 10% *1-methylamino-4-(cyanoseleno)benzene*, m. 80° (from petr. ether); $EtHN^{2}Ph$ gave, using a Pt electrode for cathode, a similar yield of the *Et deriv.*, m. 42°. G. M. Kosolapoff

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

CA

10

Organic compounds of mercury. VI. Synthesis of dialkylmercury. N. N. Mel'nikov. *J. Gen. Chem.* (U.S.S.R.) 16, 2065-71(1946)(in Russian); cf. *C.A.* 35, 6929.—Good yields of dialkyl-Hg deriva. are obtainable from alkyl halides and Na-Hg; high-percentage amalgams lower the yields of R_2Hg, with increased amt. of the Würtz reaction. The following stages are assumed: RX + Hg = RHgX; 2RHgX + 2Na = R_2Hg + 2NaX + Hg. MeBr (190 g.), 11 kg. 0.5% Na-Hg, and 74 g. MeOAc were agitated in an Fe reactor with an anchor-type stirrer at −5 to −3° for 3.5-5 hrs.; distn. with steam, followed by treatment with KOH and H_2O and redistn. gave 50-70% *Me_2Hg*, b_{760} 93-4°, d_4^{20} 2.961. The yield variations are due to imperfect sealing of the reactor and loss of MeBr. Similarly, 45 kg. 0.5% amalgam, 986 g. PrBr, and 250 cc. EtOAc gave after 4 hrs. (ice-water cooling) 69% *Pr_2Hg*, b_{20} 89-91°, d_4^{20} 2.0201, n_D^{20} 1.5162, and 59 g. hexane (from the Würtz reaction). Likewise, 550 g. iso-BuBr, 21 kg. 0.5% amalgam, and 170 g. EtOAc gave in 4 hrs. 53% *(iso-Bu)$_2$Hg*, b_{20} 108-9°, d_4^{20} 1.7670, n_D^{20} 1.4969, and 45 g. 2,5-dimethylhexane, b_{760} 107-9°, d_4^{20} 0.6990, n_D^{20} 1.3905. The prepn of Bu_2Hg was studied in more detail; all expts. were done with cooling (water temp. about 1.5°) for 4 hrs., using 0.5 mole EtOAc and 1.2 atoms Na per mol. RX; BuI gave 80-2%, BuBr 65-70%, and BuCl 11.5-17% Bu_2Hg, b_{20} 119-20°, d_4^{20} 1.7771, n_D^{20} 1.5050, with by-product octane being formed in 9-10%, 19%, and 65-70% yields, resp. A series of expts. with BuBr and amalgams of varying compn. (0.5-5%) gave gradually lower yields of Bu_2Hg (from 70 to 40%) and increased yields of octane (10 to 40%). iso-AmBr (450 g.), 17 kg. 0.5% amalgam, and 130 g. EtOAc gave, as above, 50% *(iso-Am)$_2$Hg*, b_{20} 122-5°, d_4^{20} 1.6381, n_D^{20} 1.4681, and 100 g. 2,7-dimethyloctane, b_{20} 60°, d_4^{20} 0.7282, n_D^{20} 1.4074.

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APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

CA

10

Products of the reaction between ethylene oxide and ethyl alcohol. N. N. Dolgopolov, N. N. Mel'nikov, and S. S. Nametkin. *J. Applied Chem.* (U.S.S.R.) 20, 486-93 (1947) (in Russian).—(1) The yields of Cellosolve, EtO-CH_2CH_2OH (I), Carbitol, $EtOCH_2CH_2OCH_2CH_2OH$ (II), and polyglycols (III) were detd. in the reaction between $(CH_2)_2O$ and EtOH in an autoclave, 4-24 hrs. at lower temps., 1-4 hrs. at higher temps. With 1% H_2SO_4 as catalyst, only I was formed below 85° (85 and 54% at 20-25° and 48-50°, resp.); above 200°, the products are charred or resinified; between 100° and 180°, the yields of I, II, and III are 18, 17, and 30%, resp.; the optimum temp. range is 100-30°, with the yields 49, 29, and 20%; I + II 78%, total yield 98%. At the const. temp. 120-30°, 49 g. $(CH_2)_2O$, 91 g. EtOH, the yields in 1, 2, 3, and 4 hrs. were: 40, 17, and 11; 40, 27, and 32; 42, 27, and 14; 42, 29, and 23%; the optimum length of reaction is 4-5 hrs. At 120°, increase of the molar ratio EtOH:$(CH_2)_2O$ from 1:1 to 6:1 has little effect on II but increases I (from 23 to 53%) and somewhat decreases III (from 25 to 6%); the optimum lies between 2:1 and 3:1. At 110-30°, 1-3 hrs., the optimum amt. of H_2SO_4 proved to be 0.5-1.0 g. per 77 g. $(CH_2)_2O$, further increase being without beneficial effect. Still at the const. temp. 120-30°, 2.5-3 hrs., catalysts other than H_2SO_4 proved as effective: $C_6H_4Me_2$ 3% (of the wt. of EtOH), Me_2SO_4 3%, NMe_3 11%, $MeC_6H_4SO_3H$ 2.5%. (2) I and II are in part converted into one another by heating with H_2SO_4, e.g.: 60 g. I with 1 g. H_2SO_4 at 130°, 3 hrs., gave EtOH 5 g., II 4 g., esters of III 11 g., unreacted 36 g.; 50 g. II in 8 g. EtOH, 1 g. H_2SO_4, at 105°, 5 hrs., gave EtOH 6 g., I 4 g., esters of III 10 g., unchanged 36 g. (3) I + $(CH_2)_2O$ at 120-30°, molar ratio 1:1.1 to 1:1.3, with H_2SO_4, $KHSO_4$, or Me_2SO_4 as catalysts, gave at most 70% II + III; at least 30% of the initial I remains unreacted. N. Thon.

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

CA

10

Structure and germicidal activity of organic compounds. III. Esters of betaine hydrochlorides. N. N. Mel'nikov, N. D. Sukhareva, and O. P. Arkhipova. *Zhur. Prikl. Khim.* (J. Applied Chem.) 20, 642-8(1947); cf. C.A. 40, 5668f.—A no. of [$RO_2CCHR'NMe_3R''$]X and [$RO_2CCHR'NC_5H_5$]X compds. were prepd. and tested against *Staphylococcus aureus*. The betaine hydrochloride esters were obtained from Me_3N and esters of $ClCH_2CO_2H$ by standing at room temp. The dimethylbenzylamine deriv. were prepd. by refluxing with the Cl deriv.; the pyridine deriv. were prepd. by standing several days. The percentage concn. which is germicidal is given in parentheses. $EtO_2CCH_2NMe_3Cl$, 65.7% yield (5); $PrO_2CCH_2NMe_3Cl$, 77.5% (5); $BuO_2CCH_2NMe_3Cl$, 62% (5); $AmO_2CCH_2NMe_3Cl$, 50% (5); $EtO_2CCH_2NMe_2(CH_2Ph)Cl$, 77.8%, m. 164° (from $C_6H_6Cl_2$-petr. ether) (5); $PrO_2CCH_2NMe_2(CH_2Ph)Cl$, 47.9%, m. 96° (from $C_6H_6Cl_2$-petr. ether) (5); $BuO_2CCH_2NMe_2(CH_2Ph)Cl$, 60.8%, m. 118-19° (from petr. ether) (6.5); $AmO_2CCH_2NMe_2(CH_2Ph)Cl$, 70.2%, m. 89° (from C_6H_6-petr. ether) (2); $C_6H_{11}O_2CCH_2NMe_2(CH_2Ph)Cl$, 70.3%, m. 121-2° (0.05); $C_6H_{11}O_2CCH_2NMe_2(CH_2Ph)Cl$, 72.6%, m. 131-2° (0.01); $PhO_2CCH_2NMe_2(CH_2Ph)Cl$, 39.2%, m. 144-5° (from EtOH-Et_2O) (0.5); $EtO_2CCH_2NMe_2(CH_2Ph)Br$, 24.4%, sirup (10); [$MeO_2CCH_2NC_5H_5$]Cl, 57.6%, m. 191° (2.5); [$EtO_2CCH_2NC_5H_5$]Cl, 95.7%, m. 70° (1); [$PrO_2CCH_2NC_5H_5$]Cl, 50.8%, sirup (5); [$BuO_2CCH_2NC_5H_5$]Cl, 63.6%, m. 81° (5); [$C_6H_{11}O_2CCH_2NC_5H_5$]Cl, 57.4%, m. 80° (0.01); [$PhO_2CCH_2NC_5H_5$]Cl, 62.8% (0.5); [$MeO_2CCH_2NC_5H_5$]Br, 74.6% (10); [$EtO_2CCH_2NC_5H_5$]Br, 72.7%, m. 73° (−); [$PrO_2CCH_2NC_5H_5$]Br, 64.4%, m. 111-12° (10); [$BuO_2CCH_2NC_5H_5$]Br, 61%, sirup (5); [$AmO_2CCH_2NC_5H_5$]Br, 60.6%, sirup (2). IV. **Amides and esters of betaine hydrochlorides.** *Ibid.* 21, 306-8(1948). Esters of betaine HCl salts were prepd. from $ClCH_2CO_2R$ and an equimol. amt. of amine on standing in Et_2O, EtOH, or C_6H_6, most of the products are very hygroscopic. The amides were prepd. from $ClCH_2CONH_2$ and the amines by warming in EtOH. The aromatic esters hydrolyze slowly in aq. solns. The properties of the products follow; figures in brackets indicate % conen. toxic to *Staphylococcus aureus* at 20°. $RO_2CCH_2NMe_3Cl$: R = Pr, m. 82°, 83.5% yield (0.5); C_6H_{11}, m. 88°, 28% (*chloroplatinate*, m. 210°) (0.5); C_6H_{11}, paste, 47% (*chloroplatinate*, m. 191°) (0.25); H, m. 24° (figure indistinct), 85.7% (2.5). [$RO_2CCH_2NC_5H_5$]Cl: R = C_6H_{11}, m. 85°, 65.7% (0.25); C_6H_{11}, m. 92°, 70.0% (*chloroplatinate*, m. 194°) (0.1); *p*-PhC_6H_4, m. 122°, 45.3% (0.5). [$EtO_2CCH_2N(CH_3)_2C_5H_5$]Cl, sirup, 77.8% (*chloroplatinate*, m. 220°, 138°) (1.25); [*p*-$MeC_6H_4NHCOCH_2NC_5H_5$]Cl, m. 220°, 50.4% (*chloroplatinate*, m. 218°) (5); *o*-analog, m. 182°, 55.3% (*chloroplatinate*, m. 194°) (5). [$PhNHCOCH_2NC_5H_5$]Cl, m. 228°, 67.7% (*chloroplatinate*, m. 124.5°, 57% (*chloroplatinate*, m. 83°) (0.1); *p*-$PhC_6H_4O_2CCH_2NMe_2(CH_2Ph)Cl$, m. 137-8°, 52.5% (*chloroplatinate*, m. 189°) (0.25); *p*-$PhCH_2C_6H_4O_2CCH_2NMe_2(CH_2Ph)Cl$, m. 152-3°, 85.3% (*chloroplatinate*, m. 85°) (0.25); $PhNHCOCH_2NMe_2(CH_2Ph)Cl$, m. 103°, 63.2% (*chloroplatinate*, m. 198°) (2.5); *o*-$MeC_6H_4NHCOCH_2NMe_2(CH_2Ph)Cl$, m. 180°, 48.5% (*chloroplatinate*, m. 181°).

G. M. Kosolapoff

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

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A

Halonaphthoxyacetic and halonaphthoxybutyric acids. S. S. Nametkin, N. N. Mel'nikov, and Yu. A. Baskakov. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 18, 2019-22(1948). 1,2-$ClC_{10}H_6OH$ (7 g.) and 5.6 g. KOH in 40 ml. 75% EtOH treated with 5.7 g. $ClCH_2CO_2H$ in 5 ml. H_2O and boiled 6 hrs. give the powdy sol. K salt of 1-chloro-2-naphthoxyacetic acid; free acid, m. 168.5° (from C_6H_6). Similarly 1,2-$BrC_{10}H_6OH$ gives 58% *1-bromo-2-naphthoxyacetic acid*, m. 162° (from C_6H_6); 1,6,2-$Br_2C_{10}H_5OH$ gave 66% *1,6-dibromo-2-naphthoxyacetic acid*, m. 181.5°. Refluxing 3.6 g. 1,2-$ClC_{10}H_6OH$, 1.8 g. $ClCH_2CONH_2$ and iso-AmONa (from 0.46 g. Na and 20 ml. ROH) 4 hrs. gave 70% *α-(1-chloro-2-naphthoxy)acetamide*, m. 177° (from EtOH); similarly, 1,2-$BrC_{10}H_6OH$ gave 57% *α-(1-bromo-2-naphthoxy)acetamide*, m. 188.5° (from EtOH), while 1,6,2-$Br_2C_{10}H_5OH$ gave 60% *α-(1,6-dibromo-2-naphthoxy)acetamide*, m. 218° (from EtOH). Heating the Na naphthoxides in iso-AmOH with $ClCH_2CONHPh$ 1.5 hrs. gave the following: *α-2-naphthoxyacetanilides*: *1-Cl* (88%), m. 118.5°, *1-Br* (83%), m. 129°, and *1,6-di-Br* (86%), m. 174° (from C_6H_6). To 0.69 g. Na in 25 ml. EtOH was added 0.03 mol halonaphthol and 6.5 g. $EtCHBrCO_2Et$; after refluxing 2 hrs., the mixts. were boiled 20 min. with 20 ml. 10% NaOH, another 20-30 min. with 100 ml. H_2O and charcoal, filtered, and acidified to give the following *α-2-naphthoxybutyric acids*: *1-Cl* (88%), m. 132.5° (from C_6H_6-petr. ether), *1-Br* (90%), m. 147°, *1,6-di-Br* (91%), m. 176° (from petr. ether).

G. M. Kosolapoff

ASTIA BIBLIOGRAPHICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

PA 70Y14

MEL'NIKOV, N. N.

USSR/Chemistry - Betaine Chlorohydrates Mar 1948
Chemistry - Synthesis

"The Composition and Germicidal Activity of Organic Compounds: IV. Amides and Esters of Betaine Chlorohydrates," N. N. Mel'nikov, N. D. Sukhareva, O. P. Arklipova, Chem Lab, Cen Sci Res Disinfection Inst, Ministry of Pub Health USSR, Moscow, 3 pp

"Zhur Prik Khim" Vol XXI, No 3

Series of subject compounds were synthesized and studied. (Most of these compounds have not been previously described.) Their bactericidal properties were tabulated, showing the concentration required to kill staphylococcus aureus. Submitted 1 Apr 1947.

[redacted] 70Y14

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MEL'NIKOV, N. N.

"The Chemistry of DDT" (Khimiya DDT), N. N. Mel'nikov, Goskhimizdat, Moscow/Leningrad, 1949, 120 pages, 6 rubles.

Research work done in the Scientific Institute of Fertilizers and Insecto-fungicides imeni Ya. V. Samoylov.

SO: Uspekhi Khimii, Vol 18, #6, 1949; Vol 19, #1, 1950 (W-10083)

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

MEL'NIKOV, N. N.

MEL'NIKOV, N. N., [illegible], [illegible], [illegible], [illegible]. "[illegible] of [illegible] and certain [illegible] acids in [illegible] [illegible]", [illegible] [illegible]. [illegible]-[illegible]. dezinfekts. [illegible], [illegible], [illegible], [illegible].

SO: U-[illegible]631, 16 Sept 53, ([illegible] [illegible] [illegible] Statey, No. [illegible], [illegible]).

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

Some derivatives of hydroxybiphenyl S. S. Nametkin, N. N. McFonkov, and Yu. A. Baskakov. *Zhur. Obshchei Khim.* (J. Gen. Chem.) **19**, 1151-7(1949). *p*-PhC$_6$H$_4$ONa with ClCH$_2$CONH$_2$ in iso-AmOH gave 62% *p-PhC$_6$H$_4$OCH$_2$CONH$_2$*, m. 195.6°; ClCH$_2$CONHPh similarly gave 60% *p-PhC$_6$H$_4$OCH$_2$CONHPh*, m. 149-50°. ClCH$_2$CO$_2$H (2.8 g.) in 12 ml. H$_2$O added to 4 g. 3,4-Cl(HO)C$_6$H$_3$Ph in 50 ml. NaOH and boiled 5 hrs. gave on filtration and acidification 80% (*4-phenyl-2-chlorophenoxy*)*acetic acid*, m. 100-1° (from ClCH$_2$CH$_2$Cl); *amide*, obtained in 54% yield by 0.5 hr.'s refluxing of 2,4-ClPhC$_6$H$_3$ONa with ClCH$_2$CONH$_2$ in abs. EtOH, m. 164-4.5°; *anilide*, obtained similarly from ClCH$_2$CONHPh, m. 122° (from iso-AmOH). Similar reactions with 2,4-BrPhC$_6$H$_3$OH gave 75% (*4-phenyl-2-bromophenoxy*)*acetic acid*, m. 175.5-7° (from ClCH$_2$CH$_2$Cl-petr. ether); *amide* (53%), m. 167-8.5° (from EtOH); *anilide*, m. 124° (50%). Similarly, 3,5,4-Cl$_2$HO(C$_6$H$_3$)Ph gave 71% (*4-phenyl-2,6-dichlorophenoxy*)*acetic acid*, m. 150° (from C$_6$H$_6$-petr. ether), and 75% of its *amide*, decomp. 210°; 3,5,4-Br$_2$(HO)C$_6$H$_3$Ph analogously gave 45% α-(*4-phenyl-2,6-dibromophenoxy*)*acetamide*, m. 104.5°, which (2 g.), boiled 8 hrs. with excess 10% NaOH, gave 70% of the *free acid*, m. 145-6°; *anilide* (70%), m. 140-2°. The direct prepn. of the acid was unsatisfactory. A similar procedure gave (90%) α-[*4-(4-bromophenyl)-2-chloro-6-bromophenoxy*]*acetamide*, m. 221-1.5°, which yielded (80%) of the *free acid*, m. 218°, on hydrolysis; *anilide* (35%), m. 205°. Likewise, ClCH$_2$CONH$_2$ and the corresponding Na phenoxide gave 71% α-[*4-(4-bromophenyl)-2,6-dibromophenoxy*]*acetamide*, m. 218°, which gave 42% of the *free acid*, m. 219.5-20.5° (from iso-AmOH), after 12 hrs. hydrolysis; *anilide* (50%), m. 225.5-20°. Boiling 0.8 g. 2-HOC$_6$H$_4$Ph, 7.6 g. ClCH$_2$CO$_2$H, and 60 ml. 2 N NaOH 3 hrs. gave 87% conversion (2.5 g. ROH recovery) to (*2-phenylphenoxy*)*acetic acid*, m. 109.5-10° (from C$_6$H$_6$-petr. ether), α and its salts are more sol. in H$_2$O than the 4 isomer; a similar procedure gave 70% *amide*, m. 147°, and 59% *anilide*, m. 112° (from ligroin). Similarly, 5,2-Cl(HO)C$_6$H$_3$Ph gave (*2-phenyl-4-chlorophenoxy*)*acetic acid*, m. 122° (from petr. ether), 40% of the *amide*, m. 157-7.5°, and 54% *anilide*, m. 174° (from C$_6$H$_6$-petr. ether); analogously, 3,2-Cl(HO)C$_6$H$_3$Ph gave 41% α-(*2-phenyl-6-chlorophenoxy*)*acetamide*, m. 159°, which on hydrolysis gave the *free acid* only in impure state; *anilide* (44%), m. 112°. Similarly was obtained 50.5% α-(*2-phenyl-4-bromophenoxy*)*acetamide*, m. 166° (from ligroin) which gave the *free acid* (46%), m. 137-8°; *anilide* (47%), m. 104°. Likewise, a similar condensation gave 71% α-(*2-phenyl-4,6-dichlorophenoxy*)*acetamide*, m. 143°, which gave 92% *free acid*, m. 130-30.5° (from ligroin), *anilide* (44%), m. 114.5°. 2,4,6-Cl$_3$(*p*-ClC$_6$H$_4$)C$_6$H$_2$OH analogously gave α-(*4-chlorophenyl-6,6-dichlorophenoxy*)*acetamide*, m. 162° (48%), which yielded the *free acid*, m. 169° (from ligroin), *anilide* (47%), m. 113.5° (from ligroin). Analogous reactions gave 54% α-[*2-(4-bromophenyl)-4,6-dibromophenoxy*]*acetamide*, m. 198.5°, *anilide*, m. 149-9.5°; *free acid*, m. 188° (from C$_6$H$_6$-ligroin).

G. M. K.

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MEL'NIKOV, N. N.

Feb 49

USSR/Chemistry- Paraffins, Nitro
Chemistry- Aldehydes

" Research in the Field of Aliphatic Nitrocompounds: VI, Obtaining Halogen Nitroparaffins From Oximes of Aldehydes and Ketones," Ye. M. Cherkasova, N. N. Mel'nikov, Lab Org Chem, Moscow Inst of Fine Chem Tech, 3 pp

"Zhur Obshch Khim" Vol XIX, No 2

Demonstrated that halogen nitroparaffins may be obtained by halogenation of oximes of aldehydes and ketones in an alkaline medium without preliminary separation of halogen nitroso compounds. Methods may be used for synthesis of halogen nitroparaffins oa a required structure. Submitted 22 Jun 47.

Lab. of Org. Chem., Moscow Inst. of Chem. Technology.

PA 46/49T16

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

CA

11 C

Bactericidal activity and surface tension of aqueous solutions of esters and amides of betaine hydrochlorides N. N. Mel'nikov and N. D. Sukhareva (Ministry Health, Moscow). *Zhur. Priklad. Khim.* (J. Applied Chem.) 22, 1122-3(1949); cf. *C.A.* **43**, 6976*h*.—The surface-tension lowering and bactericidal activity do not necessarily coincide, as shown in the following series. Surface tensions of aq. solns. at 20° for 1, 0.5, 0.25, and 0.125% solns., in dynes/cm. are: $[PhCH_2NMe_2(CH_2CO_2Et)]Cl$ 70.3, 70.8, 72, 72.5; Bu ester analog 62.3, 68.9, 70.7, 71.1; octyl ester analog 42.8, 50.7, 60.2, 62.8; decyl ester analog 37.1, 37.2, 40.2, 49.9; *Ph ester analog* 65.2, 69.6, 71.3, 71.9; biphenylyl ester analog (isomer unstated) 42.1, 49.6, 59.1, 68.3; *p*-benzylphenyl ester analog 41.3, 45.3, 50.1, 57.5; $Me_3N(CH_2CO_2Bu)Cl$ 64, 66.6, 68.4, 69.8; Am analog 51.4, 56.3, 60.4, 65.8; octyl analog 26.4, 38.0, 48.9, 58.2; decyl analog 22.1, 22.3, 26.7, 33.1; $C_5H_5N(CH_2CO_2C_8H_{17})Cl$ 32.7, 34.0, 42.3, 52.3; decyl analog 30.5, 33.2, 41.0, 51.1; $C_5H_5N(CHAmCO_2Et)Br$ 45.5, 53.4, 60.4, 65.6. Thus, the order of surface activity declines in the order: Me_3N^+ derivs., pyridine derivs., Me_2NCH_2Ph derivs. however, the latter are most active bactericides, and Me_3N derivs. are least effective. Cf. M., *et al.* and **21**, 306(1948).

G. M. Kosolapoff

1951

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CA

Synthesis of some alkoxyphenoxyalkylcarboxylic acids. S. S. Nametkin, N. N. Mel'nikov, and K. S. Bokarev. *Doklady Akad. Nauk S.S.S.R.* 68, 77-80(1949). -Alkoxyphenoxyacetic acids were prepd. from RONa and $ClCH_2CO_2Na$ in aq. soln.; their amides were obtained from RONa and $ClCH_2CONH_2$ in EtOH. The corresponding α-butyric deriv. were prepd. from RONa and $EtCHBrCO_2Et$ in EtOH, followed by sapon. with NaOH. The following *(alkoxyphenoxy)acetic acids* were obtained in the yields given in parentheses: *2-MeO*, m. 116-17° (66%) [*amide*, m. 138° (79%)]; *2-EtO*, m. 70-1° (58%) [*amide*, m. 128° (79%)]; *2-PrO*, m. 68° (65%) [*amide*, m. 118° (74%)]; *2-BuO*, m. 63° (68%) [*amide*, m. 102° (79%)]; *2-iso-AmO*, m. 64-5° (73%) [*amide*, m. 106° (90%)]; *2-CH_2:$CHCH_2O$*, m. 75° (62%) [*amide*, m. 90° (58%)]; *3-MeO*, m. 112° (79%) [*amide*, m. 110° (57%)]; *3-EtO*, m. 77-8° (73%) [*amide*, m. 89° (64%)]; *3-PrO*, m. 81-2° (72%) [*amide*, m. 83° (77%)]; *3-BuO*, m. 95° (74%) [*amide*, m. 93° (80%)]; *3-iso-AmO*, m. 114-15° (76%) [*amide*, m. 115° (88%)]; *3-CH_2:$CHCH_2O$*, m. 112° (57%) [*amide*, m. 138° (63%)]; *4-MeO*, m. 112° (51%) [*amide*, m. 116° (72%)]; *4-EtO*, m. 143-4° (88%) [*amide*, m. 137° (71%)]; *4-PrO*, m. 108° (68%) [*amide*, m. 135° (79%)]; *4-BuO*, m. 112° (80%) [*amide*, m. 127° (77%)]; *4-iso-AmO*, m. 124-5° (78%) [*amide*, m. 132° (75%)]; *4-CH_2:$CHCH_2O$*, m. 98-9° (53%) [*amide*, m. 114° (61%)]. *α-(Alkoxyphenoxy)butyric acids*: *2-MeO*, m. 75° (72%); *2-EtO*, m. 52° (64%); *2-PrO*, m. 82° (57%); *2-BuO*, m. 67° (66%); *2-iso-AmO*, m. 53° (63%); *2-CH_2:$CHCH_2O$*, m. 54° (61%); *3-MeO*, m. 63° (68%); *3-EtO*, m. 69° (58%); *3-PrO*, m. 75° (54%); *3-BuO*, m. 75° (58%); *3-iso-AmO*, m. 63° (67%); *4-MeO*, m. 52° (62%); *4-EtO*, m. 80° (70%); *4-PrO*, m. 66° (74%); *4-BuO*, m. 71° (58%); *4-iso-AmO*, m. 88° (65%); *4-CH_2:$CHCH_2O$*, m. 61° (67%). The products were prepd. for *bactericidal* and *fungicidal* studies.

G. M. Kosolapoff

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MEL'NIKOV, N. N.

N. N. MEL'NIKOV and Mandel'daum, Ya. A. and Popov, P. V.

"Synthesis and Insecticidal Properties of Some Esters of Phosphoric Acid", 1950

This article is the first acknowledgment in the Soviet press of compounds related to Parathion.

SO: B-65968

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

2426. IDENTIFICATION OF PHENOLS. Nametkin, SS Mel'nikov, NN Baskakov, Yu. A. and Bokarev, KS. (Zh. Anal. Khim. (J. Anal. Chem.). 1950, vol. 5, 7-13; abstr. in chem. abstr., 1950, vol. 44, 4374). Phenols are identified in the form of amides and anilides of henoxyacetic acids. Dissolve 0.1-0.2 g of Na in 8-10 ml of absolute EtOH or iso-AmOH. Add 1-1.5 g of the phenol and 0.7-1 g of chloroacetamide or 1-1.5 g of chloracetanilide. Reflux for 1-1.5 hrs, filt r while hot, cool and determine the mp. Data on 154 phenols are reported.

ASM SLA METALLURGICAL LITERATURE CLASSIFICATION

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

MEL'NIKOV, N. N.

N. N. MEL'NIKOV, A. S. Zabrodina, O. N. Kurbanov, and A. K. Ruzhentseva

"S. S. Nametkin's Work in the Field of Petroleum Chemistry", Uspkhi Khimii, Vol. XIX, No. 6, 1950, pp. 657-672

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

MEL'NIKOV, N. N.

USSR/Chemistry - Insecticides Nov 50

"From the Field of Organic Insectofungicides: X. Synthesis of Dialkyldisulfides," N. N. Mel'nikov, L. G. Vol'fson, Sci Inst of Fertilizers and Insectofungicides imeni Ya. V. Samoylov, Min of Chem Ind Lab of Org Insectofungicides

"Zhur Obshch Khim" Vol XX, No 11, pp 2085-2088

Synthesized series of aliphatic disulfides not described in literature; showed reaction of alkyl bromides with Na disulfide yields small

[redacted] 170T25

USSR/Chemistry - Insecticides (Contd) Nov 50

output of disulfides with extensively branched carbon skeleton. Small yield is due to side processes - formation of olefins.

170T25

[redacted] 170T25

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

MEL'NIKOV N.N.

USSR/Chemistry - Surface Active Compounds Nov 50

"From the Field of Organic Insectofungicides: XI. Synthesis of Iso-Alkyl Sulfonates and Their Surface Tension in Aqueous Solutions," L. G. Vol'fson, N. N. Mel'nikov, Sci Inst of Fertilizers and Insectofungicides imeni Ya. V. Samoylov, Min of Chem Ind Lab of Org Insectofungicides

"Zhur Obshch Khim" Vol XX, No 11, pp 2089-2092

Synthesized series of alkyl sulfonates not described in literature and determined surface tensions of their aqueous solutions at different concentrations.

170T26

USSR/Chemistry - Surface Active Compounds (Contd) Nov 50

Surface tensions for compounds of identical molecular weight at equal concentrations differed in dependence on structures of the compounds.

170T26

FA 170T26

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CA

Organic Chemistry - 10

Organic insectifuges. X. Synthesis of dialkyl disulfides. N. N. Mel'nikov and L. G. Vol'fson. *J. Gen. Chem. U.S.S.R.* 20, 2159-61(1950)(Engl. translation).—See *C.A.* 45, 5609d. XI. Synthesis of alkanesulfonates with iso structure and surface tension of their aqueous solutions. L. G. Vol'fson and N. N. Mel'nikov. *Ibid.* 2163-5(Engl. translation).—See *C.A.* 45, 5608g.

B. L. M.

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

CA

15 A

Synthesis and insecticidal properties of some esters of phosphoric acid. N. N. Mel'nikov, Ya. A. Mandel'baum, and P. V. Popov. *Doklady Akad. Nauk S.S.S.R.* 71, 485-7(1950).—The following esters were prepd. by unspecified and undescribed methods and were tested against *Aphis fabae* and *Calandra granaria*. The figures in parentheses indicate the min. percentage content of the ester giving 100% insect kill when used in dusting powders against the two species, resp.: $(p\text{-}O_2NC_6H_4O)_2PS(OMe)$, 51%, m. 145-7° (1.25-2.5; [illegible]); Et analog, 90%, m. 120-7° (0.1-0.25; 0.1-0.25), Pr analog, 65%, m. 92-3° (—; 5.0); iso-Pr analog, 51%, m. 96-7° (—; 5.0); Bu analog, 80%, m. 57-8° (—; 5.0); $(m\text{-}O_2NC_6H_4O)_2PS(OEt)$, 90%, m. 81-2° (—; 5.0); $p\text{-}O_2NC_6H_4OPS(OMe)_2$, 80%, d_4^{20} 1.3518, n_D^{20} 1.5600 (0.3-0.6; —); $o\text{-}O_2NC_6H_4OPS(OEt)_2$, 50%, d_4^{20} 1.2551, n_D^{20} 1.5255 (0.15-0.3; —); m-nitro analog, 79%, d_4^{20} 1.2801, n_D^{20} 1.5362 (—; 5.0); p-nitro analog, 80%, d_4^{20} 1.2704, n_D^{20} 1.5374 (0.012-0.025; 0.012-0.025); $p\text{-}O_2NC_6H_4OPS(OPr)_2$, 65%, d_4^{20} 1.2121, n_D^{20} 1.5327 (—; 5.0); diiso-Pr analog, 60%, d_4^{20} 1.2101, n_D^{20} 1.5378 (—; 5.0); di-Bu analog, 27%, d_4^{20} 1.1640, n_D^{20} 1.5311, (—; 5.0); $(p\text{-}O_2NC_6H_4O)_2PS$, 50%, m. 175-6° (above 10; above 10); $(MeO)_2PS$ (1.25-2.5; —); $(EtO)_2PS$ (1-2.5; 0.5-1.0); DDT (over 5.5; over 5.5); benzene hexachloride (3.5-7.0; 3.5-7.0). The activity of Me and Et esters is noted and the p-nitro derivs. are most potent; m-isomers are least effective. Dialkylaryl derivs. are most active, these are liquids distillable only in high vacuum.

G. M. Kosolapoff

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

1951-1956

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

MEL'NIKOV, N. N.

Selenious Oxide

Selenious oxide as an oxidizing agent in organic chemistry. Reakts. org. soed., No. 1, 1951.

Monthly List of Russian Accessions, Library of Congress, December 1952. UNCLASSIFIED

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

MEL'NIKOV, N.N., [illegible], V.M., [illegible], A.N., [illegible], A.S.

"Academician Sergey Semenovich Nametkin" Zhur Obshch Khim No 12 Dec 1951 pp 2101- 2146 w-[illegible]

APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

p

CA

p-Alkoxyphenoxyacetic acids and their simple derivatives. S. S. Nametkin, N. N. Mel'nikov, and K. S. Bokarev. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 327-30(1951).—$ClCH_2CO_2Na$ with *p*-$MeOC_6H_4ONa$ in H_2O gave 51% (*p-methoxyphenoxy*)*acetic acid*, m. 112°. Refluxing 0.92 g. Na in 40 ml. abs. EtOH with 4.96 g. *p*-$MeOC_6H_4OH$ and 3.74 g. $ClCH_2CONH_2$ gave 71.8% *α*-(*p-methoxyphenoxy*)*acetamide*, m. 116° (from H_2O). Heating 16 g. *p*-$EtOC_6H_4OH$, 100 ml. 10% NaOH, and 11.3 g. $ClCH_2CO_2H$ 1 hr. in 20 ml. H_2O, acidification, extn. with Et_2O, washing the ext. with satd. Na_2CO_3, and treating the ppt. and the Et_2O soln. with HCl gave 88% (*p-ethoxyphenoxy*)*acetic acid*, m. 143-4° (from H_2O); its soly. is 2.2 g. in 100 ml. H_2O; its *amide*, m. 137°, was obtained as above in 70.5% yield. The following (*4-alkoxyphenoxy*)*acetic acids* were similarly prepd.: *propoxy* (67.6%), m. 108°; soly. 0.1 g. 100 ml. H_2O; *amide*, m. 134.5° (from 40% MeOH); prepd. in 79% yield; *butoxy* 80%, m. 11[illegible] (from 50% MeOH); [*amide*, m. 126.7° (from 80% MeOH) formed in 76.4% yield]; *isoamoxy* (78%), m. 124° (from 50% MeOH); [*amide*, m. 142° (from 60% MeOH) obtained in 75% yield]. To EtONa from 1.15 g. Na in 30 ml. EtOH were added 4.96 g. *p*-$MeOC_6H_4OH$ and 7.84 g. $EtCHBrCO_2Et$, and the mixt. boiled 2 hrs., addl. of [illegible] ml. 10% NaOH, boiling 10 min., and the usual treatment yielded *α*-(*p-methoxyphenoxy*)*butyric acid*, m. 52° (from petr. ether). By this method, the *p-EtO* [illegible], m. 79.8°, formed in 69.2% yield; the *p-propoxy* [illegible], m. [illegible], in [illegible]2% yield, and the *p-butoxy* [illegible], m. [illegible], formed in [illegible]7.4% yield (33.7% when aq. NaOH was used in the condensation). The *4-isoamoxy* [illegible], m. 88°, formed in [illegible]5% yield in EtOH soln. as above, or in [illegible] yield in aq. medium. [illegible] M. Kosolapoff

1957

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MEL'NIKOV, N. N. 176T18

USSR/Chemistry - New Chemotherapeutic Agents Feb 51

"4-Alkoxy-Phenoxyacetic Acids and Their Simplest Derivatives," S. S. Nametkin, N. N. Mel'nikov, K. S. Bokarev

"Zhur Obshch Khim" Vol XXI, No 2, pp 327-330

4-Alkoxy-phenoxyacetic acids are of interest due to activity in relation to acid-resistant microorganisms. Synthesized 4-ethoxy, 4-propyloxy-, 4-butyloxy, 4-isoamyloxy-phenoxyacetic acids and amines, and methoxy-, ethoxy-, propyloxy-, butyloxy-, and isoamyloxy- -butyrich acids (non described) with very satisfactory yields. Submitted 15 Mar 48.

PA 176T18

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C. A.
1951

Organic chemistry
10

Mechanism of oxidation of organic compounds by selenic anhydride. XII Oxidation of naphthaleneacetonitrile and esters of naphthaleneacetic acid. N. N. Mel'nikov and Yu. A. Baskakov (All-Union Mendeleev Soc., Moscow). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 604-6(1951); cf. *C.A.* 40, 5702f.—Oxidation by SeO_2 of 1-$C_{10}H_7CH_2CN$ and esters of $C_{10}H_7CH_2CO_2H$ goes by the route of oxidation of the methylene group to CO. Heating 16.7 g. 1-$C_{10}H_7CH_2CN$ and 11.1 g. powd. SeO_2 to 180°, followed, after subsidence of reaction, by 20 min. at 180-200°, gave some HCN, 78% Se, 24% unreacted nitrile, and 47% 1-$C_{10}H_7COCN$, m. 100-1°, while acidification of the mother liquor gave 22% 1-naphthoic acid, m. 158°. Heating 5.6 g. SeO_2 with 10 g. 1-$C_{10}H_7CH_2CO_2Me$ 1.5 hr. at 190° gave 92.3% Se and 68% 1-$C_{10}H_7COCO_2Me$, bp 201-5°, m. 73° (picrate, m. 81°), as well as 14% 1-$C_{10}H_7CO_2H$. The Et ester gave 72% 1-$C_{10}H_7COCO_2Et$, bp 210-12° (picrate, m. 70-6.5°).

G. M. Kosolapoff

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MEL'NIKOV N. N. ~~Nov~~. 51
Dec

USSR/Chemistry - Petroleum
Herbicides

"Academician Sergey Semenovich Nametkin," V. M. Rodionov, A.K. Ruzhentsova, A. S. Nekrasov, N. N. Mel'nikov

"Zhur Obshch Khim" Vol XXI, No 12, pp 2101-2146

Briefly reviews life and professional activities of Acad Nametkin (1876 - 1950), with summaries of his major achievements in different fields of chemisty, including nitration of hydrocarbons, petroleum chemistry, organometallic compds, plant growth stimulants, and herbicides. Includes comprehensive bibliography of nametkin's published works.

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MEL'NIKOV, N. N.

S. S. Nametkin (dead), Yu. A. Baskakov and N. N. Mel'nikov

"Synthesis of Certain Alkyl- and Aralklyphenoxyacetic Acids and Their Derivatives," Zhurnal Obshchey Khimii, Vol. XXI, No. 12, 1951, pp 2146-2150

A reference to herbicides is noted in an article in the periodical Khimicheskaya Promyshlennost', No. 1, 1951, p. 20, by N. N. Mel'nikov, in which was discussed the large-scale use, as herbicides, of different substituted phenoxyacetic acids, especially 2,4-dichlorophenoxyacetic acid, 2-methyl-4-chlorophenoxyacetic acid, and their salts.

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NAMETKIN, S.S.; BOKAREV, K.S.; MEL'NIKOV, N.N.

Synthesis of certain ethers of halohydrinquinones and halohydrincatechins. Doklady Akad.nauk SSSR 77 no.2:293-296 11 Mar 51.(CLML 20:6)

1. Academician S.S. Nametkin, deceased.

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

NAMETKIN, S.S.; BOKAREV, K.S.; MEL'NIKOV, N.N.

Synthesis of certain alkoxyhaloidphenoxyusnic acids and their derivatives. Doklady Akad. nauk SSSR 77 no.3:419-422 21 Mar 1951. (CIML 20:7)

1. Institute of Plant Physiology imeni K.A. Timiryazev of the Academy of Sciences USSR. 2. Academician S.S. Nametkin, deceased.

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MEL'NIKOV, N. N.

CATALYSTS

Chemical Abst.
Vol. 48 No. 3
Feb. 10, 1954
Organic Chemistry

(3) chem

Nametkin, S. S., Rodionov, V. M., and Mel'nikov, N. N., Reaktsii i Metody Issledovaniya Organicheskikh Soedinenii (Reactions and Methods of Examination of Organic Compounds). Moscow: Goskhimizdat, 1952. Reviewed in Uspekhi Khim. 21, 1526-7(1952).

8-31-54

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

MEL'NIKOV, N. N.; MANDEL'BAUM, Ya. A.; SVENTSITSKIY, Ye. I.

"The New Insecticide Diethyl-4-nitrophenylthiophosphate (NIUIF-100)," Khimicheskaya Promyshlennost', No 9, 1952, pp 1-3.

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R00103

MEL'NIKOV, N.N.; BASKAKOV, Yu.A.

Mechanism of oxidation of nitriles by selenium dioxide. Doklady Akad. Nauk S.S.S.R. 85, 337-9 '52. (MLRA 5:8)
(CA 47 no.15:7461 '53)

Established that when an excess of SeO_2 is used with some nitriles, a new nitrile containing one less carbon atom is formed from the initial acyl cyanide. This reaction was used to oxidize o-chlorophenylaceto nitrile, m-chlorophenylaceto nitrile, p-chlorophenylaceto nitrile, and a-naphthylaceto nitrile and to prepare the lower nitriles from them.
Presented by Acad. V.M.Rádionov 8 May 52 256T5

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"APPROVED FOR RELEASE: Wednesday, June 21, 2000 CIA-RDP86-00513R001033

USSR/Chemistry - Organophosphorus Compounds 21 Sep 52

"Synthesis of Some Mixed Esters of Dithiophosphoric Acid," N. N. Mel'nikov, K. D. Shvetsova-Shilovskaya, Sci Inst of Fertilizers and Insectofungicides

DAN SSSR, Vol 86, No 3, pp 543-545

Mixed esters of thiophosphoric acids have received considerable attention during recent years because they are effective insecticides. Some esters of dithiophosphoric acids are also of interest from this standpoint, although substances of this class are on the whole less active. In the present work, 31

247T5

dithiophosphoric acid esters are described. They were prepd by reacting dialkyldithiophosphoric acids with olefins. The initial dialkyldithiophosphoric acids were prepd by reacting the corresponding alcs with phosphorus pentasulfide. Presented by Acad V. M. Rodionov 21 Jul 52

Translation W-27612, 31 Aug 53

247T5

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MEL'NIKOV, N.N.

Synthesis of some indole derivatives. Yu. A. Bankovy and N. N. Mel'nikov (K. A. Timiryazev Inst. Plant Physiol., Acad. Sci. U.S.S.R.). *Sbornik Statei Obshchei Khim., Akad. Nauk S.S.S.R.* 1, 712-13(1953).—Ketones of the indole series which have an unsubstituted H atom at the N atom do not give normal products of the Willgerodt reaction. To 14 g. 3-acetylindole and 30 g. NaOH in 300 ml. H_2O was added over 1 hr. 63 g. Me_2SO_4 at 80-5°, stirred 4 hrs. and allowed to stand overnight, filtered and the ppt. washed free of alkali, taken up in C_6H_6 and the soln. cooled, filtered and evapd., yielding 95% *1-methyl-3-acetylindole* (I), m. 95°. Similarly were obtained: 98% *1-ethyl-3-acetylindole*, m. 88°; 100% *1-benzoyl-3-acetylindole* (II), m. 119°. Heating 5 g. I in 20 ml. dioxane with 25 ml. aq. NH_4 polysulfide in an ampul 4 hrs. at 165-71° gave 33% *1-methylindolyl-3-acetamide*, m. 181°, which boiled 5-6 hrs. with 15% KOH gave *1-methylindolyl-3-acetic acid*, m. 123°. Similarly were obtained: 89.6% *1-ethylindolyl-3-acetamide*, m. 158°, which gave the free acid, m. 108°; 60% *1-methylindolyl-2-acetamide* (from 1-methyl-2-acetylindole), m. 161.5°, which gave the free acid, decomp. 185-7.0°. II failed to yield a corresponding amide.

G. M. Kosolapoff

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Melnikov, N.N.

Analytical Abst.
Vol. 1 No. 3
Mar. 1954
Organic Analysis

(2) Chem

515. Determination of esters of phenylcarbamic acid. Yu. A. Bashakov and N. N. Melnikov (J. Anal. Chem., U.S.S.R., 1953, 8, [illegible]).—Hydrolysis of methyl and ethyl esters of phenylcarbamic acid by means of aq. alcoholic KOH and of isopropyl esters of phenylcarbamic acid and 3-chlorophenylcarbamic acid by boiling with conc. HCl yields aniline or the corresponding amine, which can be titrated with $NaNO_2$ soln.

G. S. Smith

(CA 47 no. 20: 10410 '53)

Inst. Plant Physiol. im. Timiryazev

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MEL'NIKOV, N. N.

Chem Abs v48
1-25-54
Pesticides

Organic compounds of phosphorus as insecticides. N. N. Mel'nikov. *Uspekhi Khim.* 22, 253-78(1953).—Review with 200 references. G. M. Kosolapoff

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