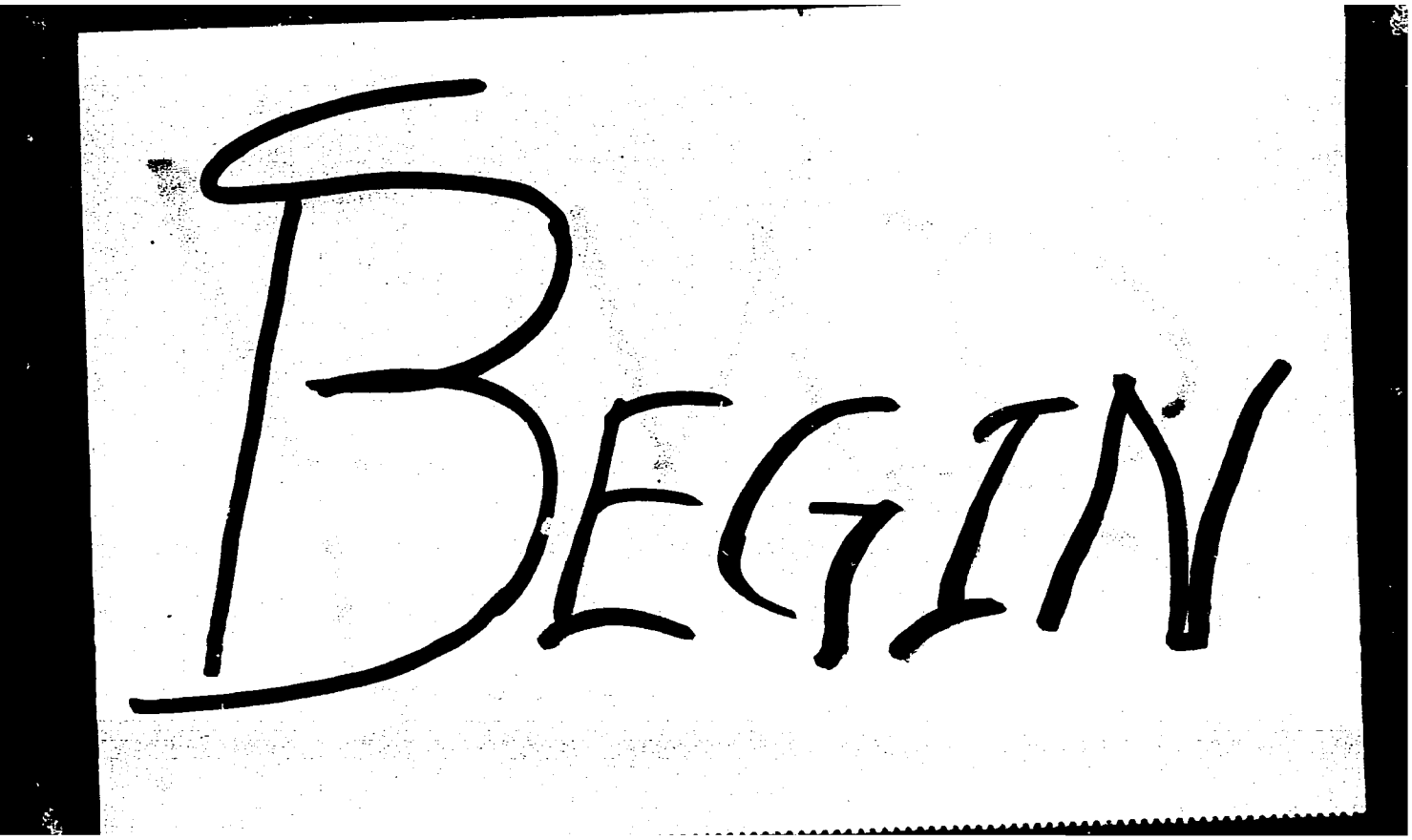


APPROVED FOR RELEASE: 06/08/2000 CIA-RDP86-00513R000205010001-2"

L 39556-66 GD
ACC NR: AT6008783 SOURCE CODE: UR/2657/65/000/014/0020/0050

AUTHOR: Berlin, A. S.

B+1

ORG: none

TITLE: Selecting the electrophysical parameters of semiconductor materials intended for diodes used in SHF cooled amplifiers

SOURCE: Poluprovodnikovyye pribory i ikh primeneniye; sbornik statey, no. 14, 1965, 20-50

TOPIC TAGS: SHF amplifier, solid state amplifier, semiconductor diode semiconducting material

ABSTRACT: Based on 1959-65 Soviet and 1960-64 Western publications, this review-type article considers parameters of Si, Ge, GaAs, InSb from the viewpoint of using these materials in liquid-helium-temperature parametric diodes.

Card 1/2 UDC: 621.382.28

L 39556-66
ACC NR: AT6008783

In a single-stage diode, the noise temperature decreases with the diode temperature, provided the diode parameters do not vary. These parameters of semiconductor materials are considered: maximum Q-factor, electric strength of the junction, majority-carrier concentration in the base, majority-carrier mobility, working-temperature range. Best published diode characteristics are tabulated. An n-GaAs source material with $N > 10^{17}$ per cm^3 is recommended for cooled diodes. Formulas and curves are presented which connect GaAs sharp-junction homogeneous-base diode parameters with the parameters of material and the geometry of contact. Also, design of a diffusion-type GaAs diode functioning at room temperature is given, as are formulas for calculating the effect of temperature on diode parameters. With proper selection of parameters, GaAs permits building high-quality point-contact and diffusion cooled-type diodes for the SHF-band operation. Orig. art. has: 9 figures, 33 formulas, and 3 tables.

SUB CODE: 09 / SUBM DATE: none / ORIG REF: 012 / OTH REF: 025

Card 2/2 fvs

L 5143-66 EWT(d)/EWT(l)/EWA(h)
ACCESSION NR: AP5026910

UR/0109/65/010/010/1907/1909
621.375.933.029.65

AUTHOR: Berlin, A. S.; Vizel', A. A.; Vystavkin, A. N.; Popov, Ye. I.; Khotuntsev, Yu. L.; Shtykov, V. D. 34 B

TITLE: Parametric amplification in the 8-mm band 25

SOURCE: Radiotekhnika i elektronika, v. 10, no. 10, 1965, 1907-1909

TOPIC TAGS: parametric amplification, millimeter wave 4

ABSTRACT: In recently published articles (B. C. DeLoach, Proc. IEEE, 1963, 51, 8, 1153 and others) on millimeter-band semiconductor amplifiers, no characteristics have been reported. The present article describes the design and characteristics of and indicates an application for an 8-mm-band parametric amplifier. Coaxial-design epitaxial germanium diodes with 0.04—0.08-pf capacitance and 3—5-v reverse voltage were used in most experiments; critical frequency at a bias of -3 v was 280—430 Gc. The diodes operated as amplifiers at a low pumping power and an operating-point bias of 0.5—2 v. The diodes were tested within -60 + 85C; up to +60C, the leakage current at -1.5 v was 1 μamp or less. The new diodes were tested in a single-cavity 8-mm parametric amplifier (see Fig. 1 of Enclosure). The signal is applied via a tapered waveguide matching unit 1. Behind the diode 4, a short-circuiting section 2 is arranged whose length equals an odd number of

Card 1/3

L 5143-66
ACCESSION NR: AP5026910

quarter-waves. The amplifier is tuned by a short-circuiting line 3 that has a characteristic resistance of 100 ohm. Transformer 5 serves for adjusting the coupling. With a gain of 20 db, the passband was 78 Mc and the noise temperature, 600 ± 150K. The parametric amplifier was used in a modulation-type radiometer whose fluctuation sensitivity was measured. Orig. art. has: 3 figures and 2 formulas.

[03]

ASSOCIATION: none

SUBMITTED: 23Jan65 ENCL: 01 SUB CODE: EC.

NO REFO SOV: 002 OTHER: 003 ATD PRESS: 4134

Card 2/3

L 5143-66
ACCESSION NR: AP5026910

ENCLOSURE: 01

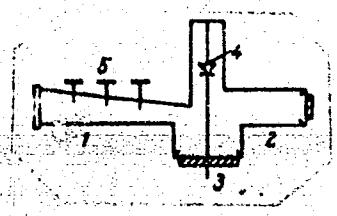


Fig. 1. A parametric semiconductor amplifier for the 8-mm band

Card 3/3

L 7792-66

ACC NR: AP5027633 SOURCE CODE: UR/0109/65/010/011/2081/2084

AUTHOR: Berlin, A. S.; Davydov, V. M.

ORG: none

TITLE: Method for measuring the Q-factor of nonlinear-capacitance diodes at shf which does not require reference standards or tuning of the measuring chamber

SOURCE: Radiotekhnika i elektronika, v. 10, no. 11, 1965, 2081-2084

TOPIC TAGS: semiconductor diode, shf measurement

ABSTRACT: Regarding a negative-bias diode as a passive linear quadripole, a new formula is developed which permits determining the Q-factor of the diode active region on the basis of measured voltage standing-wave ratio and phase shift at two bias voltages in any measuring chamber, without resistance reference standards. The spread of diode-case parameters does not affect the accuracy of measurements. An experimental verification of the formula is claimed. The method is recommended for 10—100-Gc band and for the cases when retuning of the diode chamber is undesirable. Orig. art. has: 3 figures and 21 formulas.

SUB CODE: 09 / SUBM DATE: 01Feb65 / ORIG REF: 002 / OTH REF: 001

nw

Card 1/1

UDC: 621.317.337:621.382.2

AUTHOR: Berlin, A.Ya., Engineer SOV-91-58-4-13/29

TITLE: Grounding Blades in 6/35 kv Covered Distributing Centers (Zazemlyayushchiye nozhi v zakrytykh raspredeli'tel'nykh ustroystvakh 6-35 kv)

PERIODICAL: Energetik, 1958, Nr 4, pp 18-19 (USSR)

ABSTRACT: The author suggests the use of Y-shaped grounding blades for 6/35 kv covered distributing centers with vertical bar disconnectors having mechanical blocking system. This mechanical blocking system permits the grounding blades to be switched on only if both bar disconnectors are switched off. These blades, installed on the fork of the 6 kv bar disconnector, have a precise operation and their design can be utilized for bar disconnectors with ordinary bar systems and for 6/35 kv line disconnectors.
There is 1 diagram and 1 Soviet reference.

1. Electrical networks--Equipment 2. Disconnect fittings --Equipment

Card 1/1

BERLIN, A.Ya.; MAKAROVA, A.N.

Some reactions of bis(β-hydroxyethyl)amino-ρ-benzoquinone. Part 2. Zhur. ob. khim. 30 no.11:3718-3721 N'60. (MIRA 13:11)

1. Institut eksperimental'noy i klinicheskoy onkologii AMN SSSR.
(Benzoquinone)

BERLIN, A.Ya.; KURDYUMOVA, K.N.

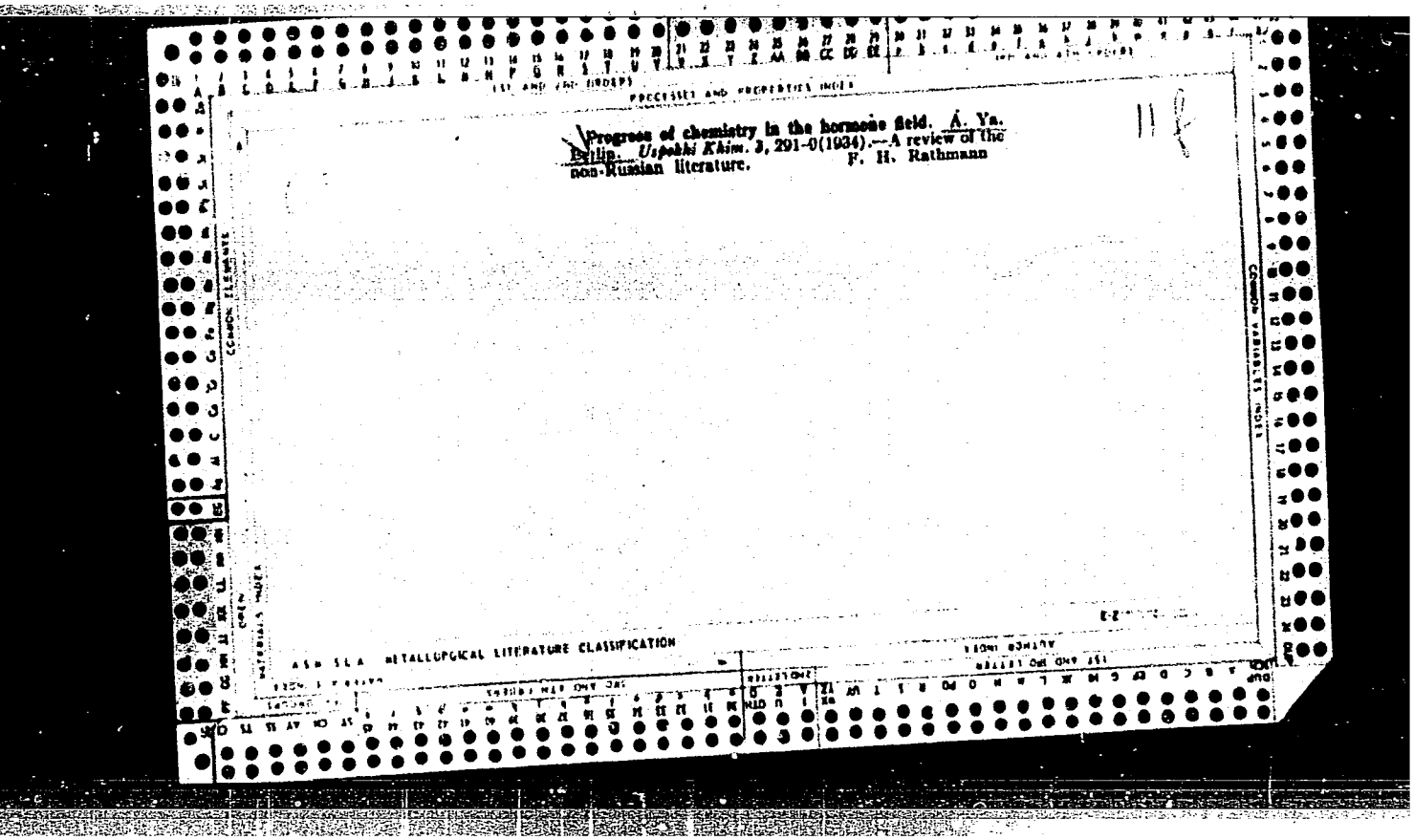
Synthesis of p-diazoacetyl derivatives of phenylalanine. Zhur. ob. khim. 30 no.11:3759-3766 N'60. (MIRA 13:11)
(Alanine)

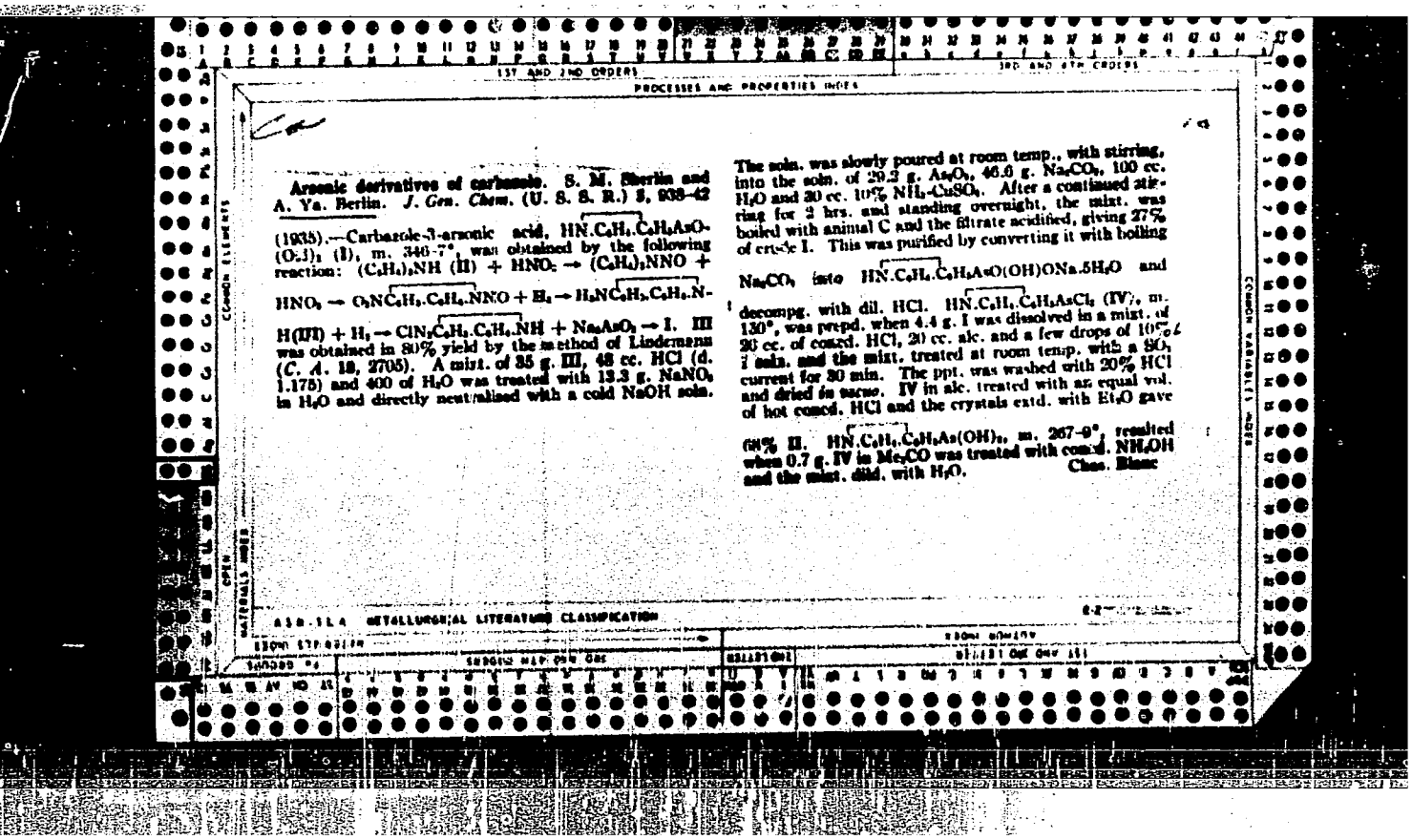
1ST AND 2ND ORDERS — PROCESSES AND PROPERTIES INDEX — 3RD AND 4TH ORDERS

10

Arsino sulfides of the fatty and aromatic series. A. B. Kretov and A. Ya. Berlin.
J. Gen. Chem. (U. S. S. R.) 1, 411-8(1931).—Bayer (*Ann.* 107, 279(1858)) and Schulte (*Ber.* 15, 1955(1882)), and later Nametkin and Nekrasov (*C. A.* 23, 3874) described the prepn. of org. arsino sulfides by the action of H_2S on arsino chlorides or the oxides. A series of fatty and aromatic arsino sulfides obtained by K. and B. are either new compds. or were prepd. by novel methods. The results of the investigation fully support the findings of Blicke and Smith (*C. A.* 24, 4002) that the org. arsino sulfides of the formula $(R_2As)_2S$ are monomers with normal mol. wt., while those of the formula RAs:S are polymers with abnormal mol. wt. The action of KSH and Na_2S on arsino chlorides in alc. produces identical compds. with those obtained with H_2S. *β-Chlorovinylarsino sulfide*, ClCH:CHAsS, was obtained in 1.6-g. yield by passing dry H_2S into 3 g. of β-ClCH:$CHAsCl_2$ in abs. alc., then bringing the reaction mass to a boil and quickly cooling, light yellow plates with repulsive odor, sol. in CS_2 and $CHCl_3$, poorly sol. in alc. *β-Hydroxyvinylarsino sulfide*, HOCH:CHAsS, obtained in 0.1-g. yield by passing H_2S into 2 g. of β-HOCH:$CHAsCl_2$ in alc. in the presence of $BaCO_3$, filtering, adding H_2O to formation of a turbidity, allowing to stand 24 hrs., sepg. the crystals from the viscous ppt. and washing with a little Me_2CO, m. 66-70°. *β-Acetoxyethylarsino sulfide*, $AcOC_2H_4AsS$, prepd. from 2 g. of $AcOC_2H_4AsCl_2$ in alc., $BaCO_3$ and H_2S, m. 69°, sol. in alc., insol. in Et_2O and $CHCl_3$. *Ethylarsino sulfide* was prepd. by Den by a different method (cf. Tolman, *C. A.* 5, 15, and Washburn, *C. A.* 12, 559). By the interaction of H_2S and 65 g. of $EtAsCl_2$ in alc. was formed an oil, which was sepd. and dissolved in $CHCl_3$, then dried over $CaCl_2$, and evapd. *in vacuo*, giving 45 g. yellow oil with offensive odor, sol. in CS_2 and $CHCl_3$, $d_{17.4}$ 1.8218, mol. wt. found 809.1, calcd. 810. *Phenylarsino*

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

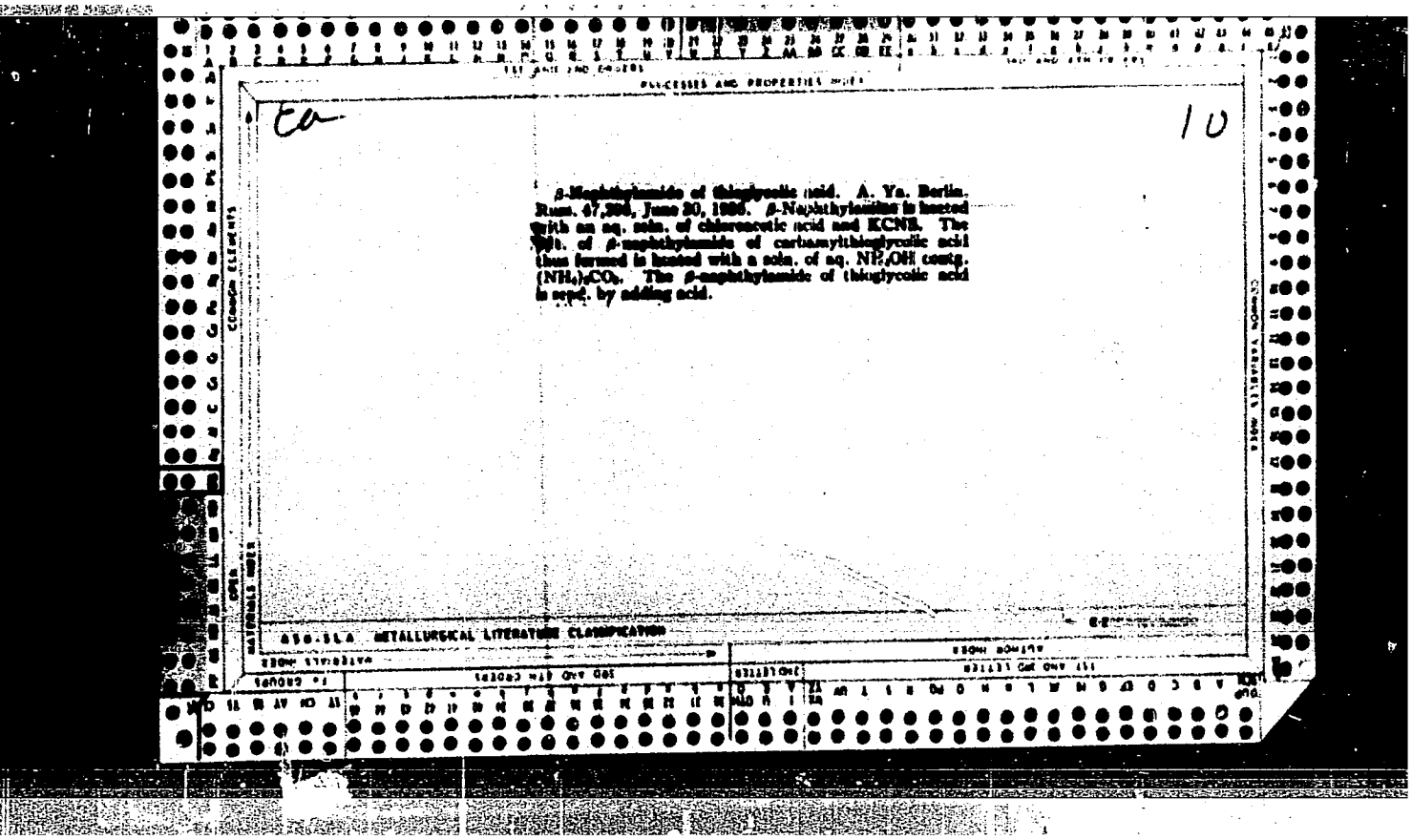




PROCESSES AND PROPERTIES INDEX

Arsenic derivatives of carbazole. S. M. Sherlin and A. Ya. Berlin. *J. Gen. Chem.* (U. S. S. R.) 5, 938–42 (1935).—Carbazole-3-arsonic acid, $HN.C_6H_3.C_6H_3AsO(O_2)_2$ (I), m. 340–7°, was obtained by the following reaction: $(C_6H_5)_2NH$ (II) + HNO_2 → $(C_6H_5)_2NNO$ + HNO_3 → $O_2NC_6H_3.C_6H_4.NNO + H_2$ → $H_2NC_6H_3.C_6H_4.NH$ (III) + H_2 → $ClN_2C_6H_3.C_6H_4.NH + NaAsO_2$ → I. III was obtained in 80% yield by the method of Lindemann (*C. A.* 18, 2705). A mixt. of 35 g. III, 48 cc. HCl (d. 1.178) and 400 of H_2O was treated with 13.3 g. $NaNO_2$ in H_2O and directly neutralized with a cold NaOH soln. The soln. was slowly poured at room temp., with stirring, into the soln. of 29.2 g. As_2O_3, 46.6 g. Na_2CO_3, 100 cc. H_2O and 20 cc. 10% NH_3-$CuSO_4$. After a continued stirring for 2 hrs. and standing overnight, the mixt. was boiled with animal C and the filtrate acidified, giving 27% of crude I. This was purified by converting it with boiling Na_2CO_3 into $HN.C_6H_3.C_6H_3AsO(OH)ONa.5H_2O$ and decompg. with dil. HCl. $HN.C_6H_3.C_6H_3AsCl_2$ (IV), m. 130°, was prepd. when 4.4 g. I was dissolved in a mixt. of 20 cc. of concd. HCl, 20 cc. alc. and a few drops of 10% I_2 soln. and the mixt. treated at room temp. with a SO_2 current for 30 min. The ppt. was washed with 20% HCl and dried *in vacuo*. IV in alc. treated with an equal vol. of hot concd. HCl and the crystals extd. with Et_2O gave 68% II. $HN.C_6H_3.C_6H_3As(OH)_2$, m. 267–9°, resulted when 0.7 g. IV in Me_2CO was treated with concd. NH_4OH and the mixt. dild. with H_2O. Chas. Blanc

A 38-11 4 METALLURGICAL LITERATURE CLASSIFICATION

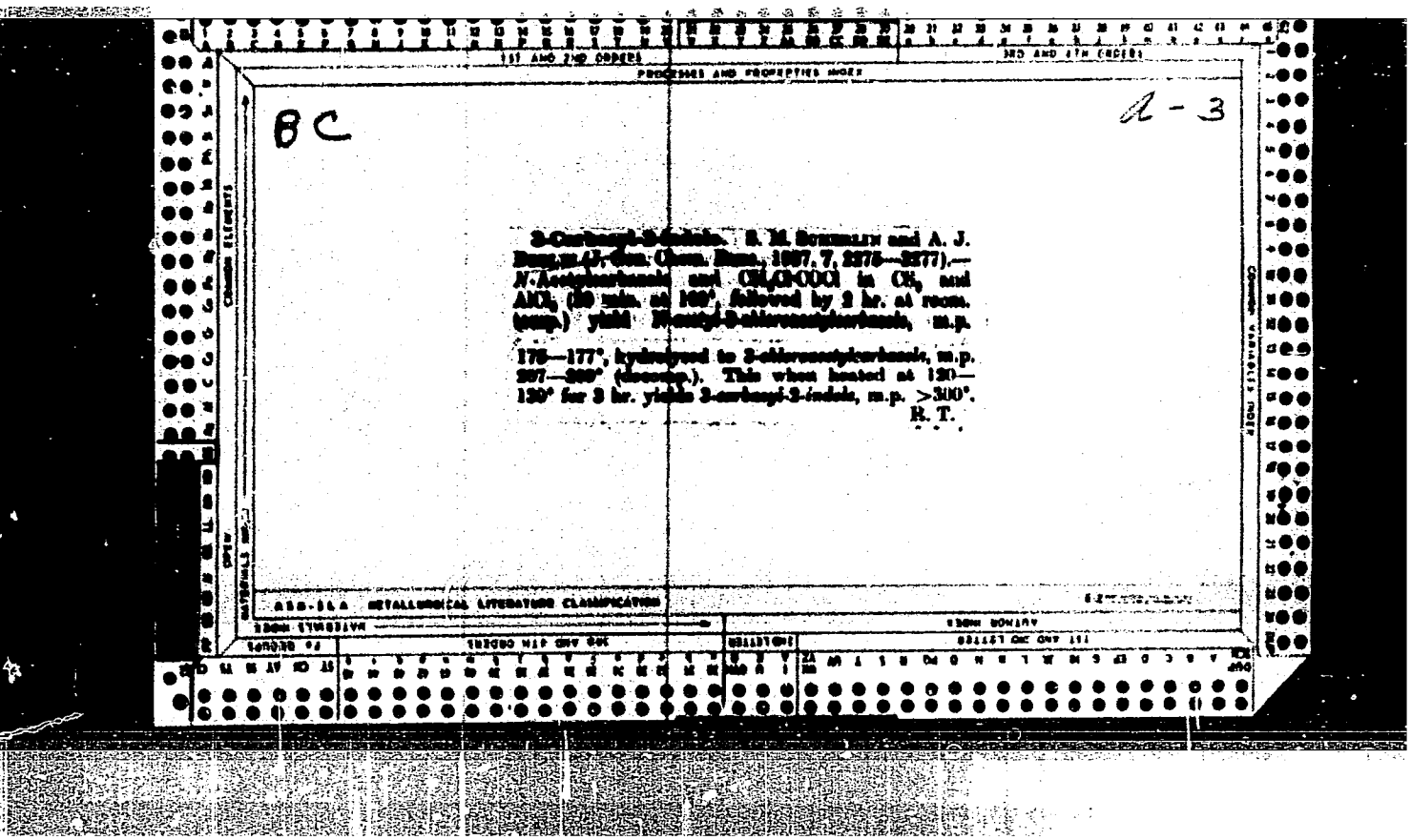


Ea

10

β-Naphthylamide of thioglycolic acid. A. Ya. Berlin. Russ. 47,306, June 20, 1955. β-Naphthylamine is heated with an aq. soln. of chloroacetic acid and KCNS. The ppt. of β-naphthylamide of carbamylthioglycolic acid thus formed is heated with a soln. of aq. NH_2OH contg. $(NH_4)_2CO_3$. The β-naphthylamide of thioglycolic acid is sepd. by adding acid.

A50-SLA METALLURGICAL LITERATURE CLASSIFICATION

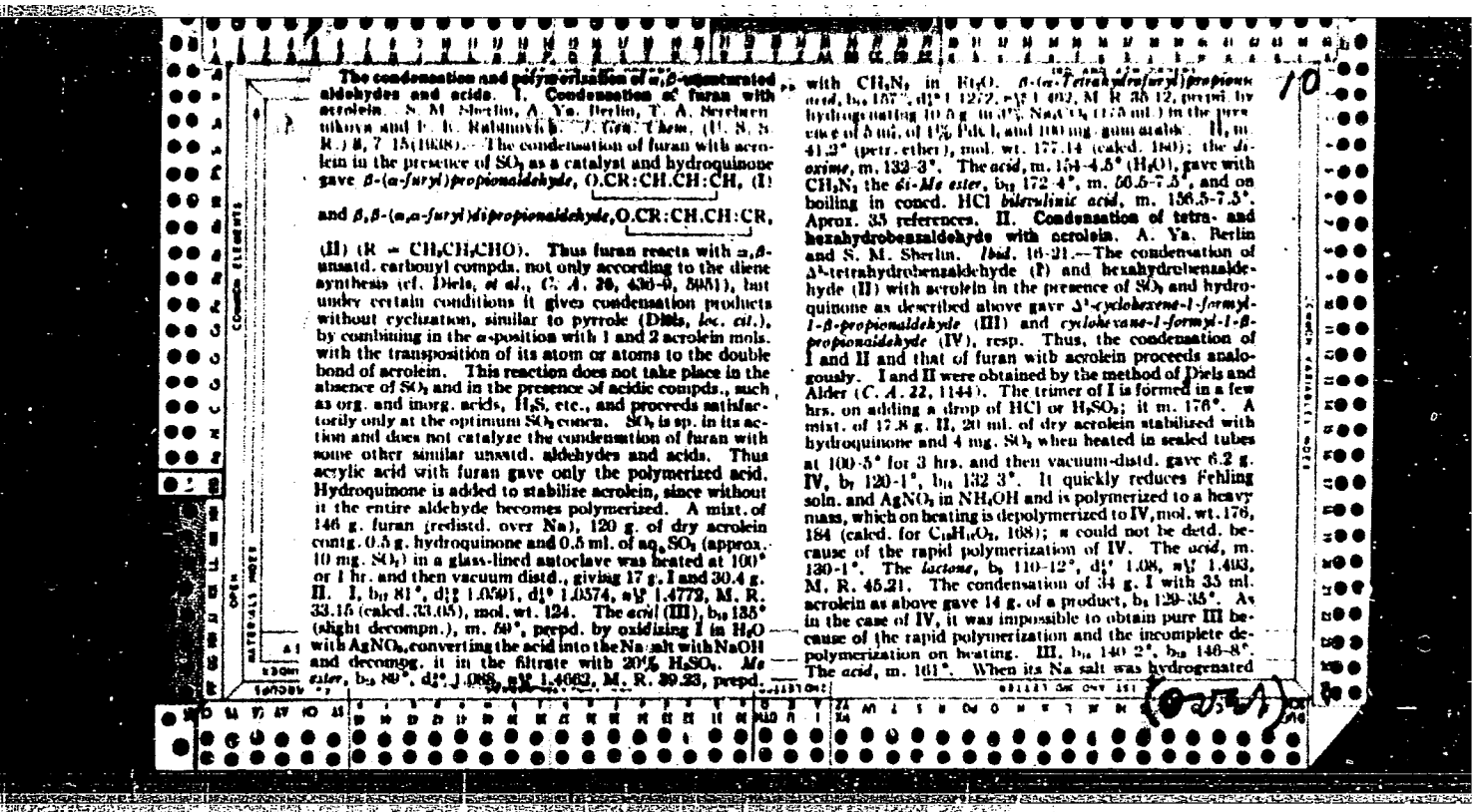


BC

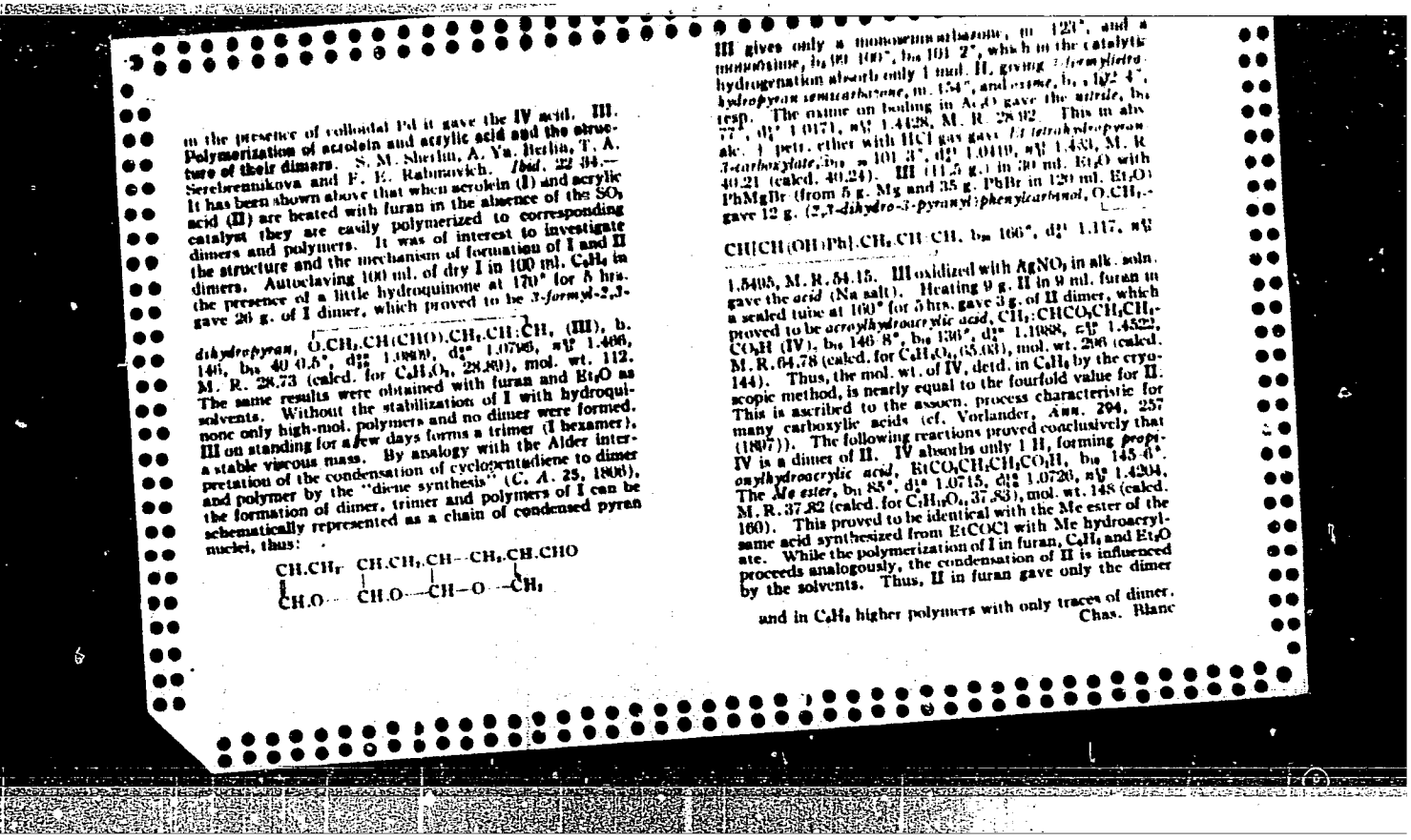
A-3

3-Carbonyl-2-indole. R. M. Bowman and A. J. Rung, J. Am. Chem. Soc., 1957, 7, 2276—2277).—*N*-Acetylcarbanole and $CH_2ClCOCl$ in CS_2 and $AlCl_3$ (30 min. at 10°, followed by 2 hr. at room temp.) yield 3-carbonyl-2-chloroacetylcarbanole, m.p. 176—177°, hydrolysed to 3-chloroacetylcarbanole, m.p. 207—209° (decomp.). This when heated at 120—130° for 3 hr. yields 3-carbonyl-2-indole, m.p. >300°.

R. T.



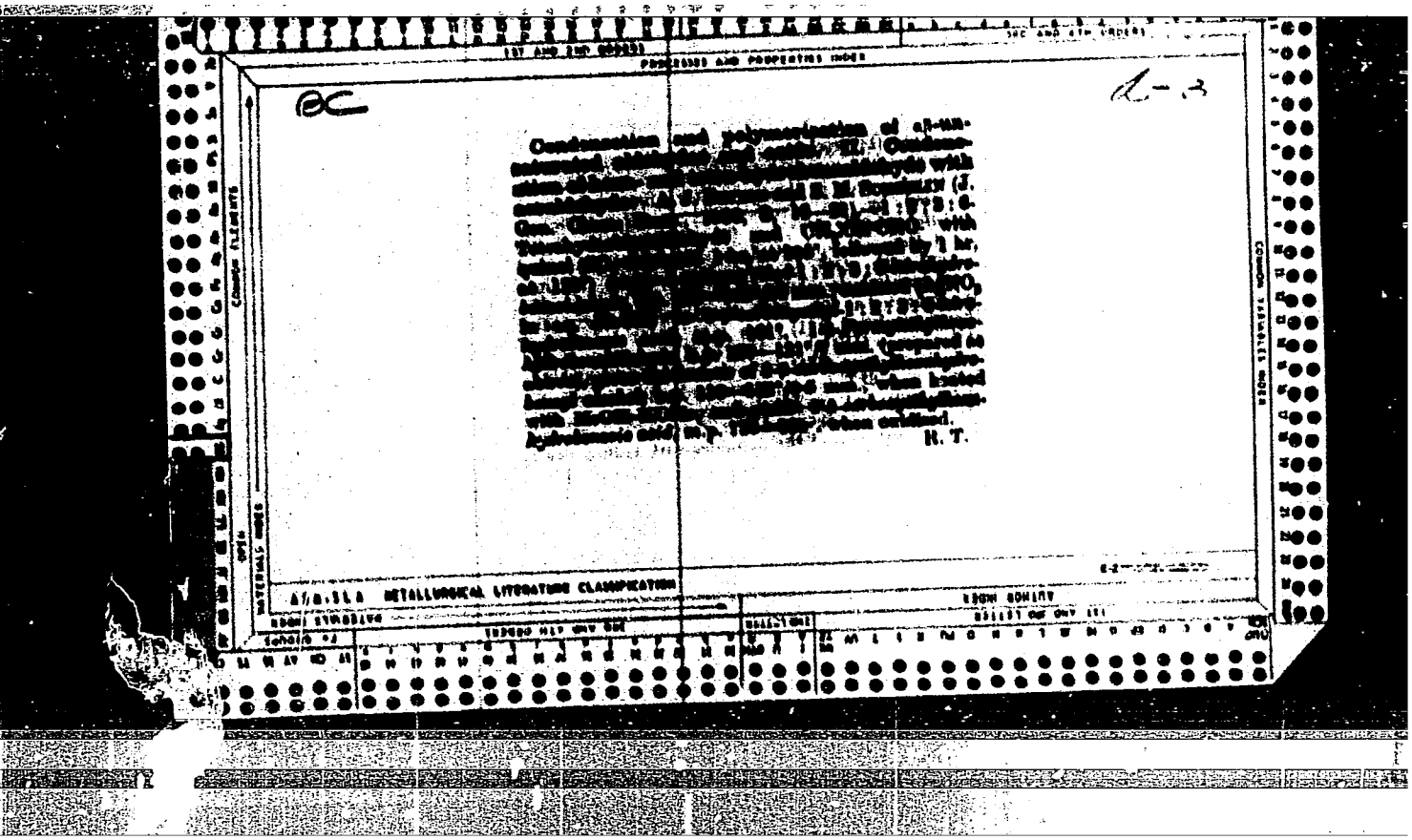
The condensation and polymerization of α,β-unsaturated aldehydes and acids. I. Condensation of furan with acrolein. S. M. Sherlin, A. Ya. Berlin, T. A. Serebrennikova and P. D. Rabinovich. *J. Gen. Chem. (U. S. S. R.)* **8**, 7-15(1938).—The condensation of furan with acrolein in the presence of SO_2 as a catalyst and hydroquinone gave *β-(α-furyl)propionaldehyde*, O.CR:CH.CH:CH, (I) and *β,β-(α,α-furyl)dipropionaldehyde*, O.CR:CH.CH:CR, (II) (R = CH_2CH_2CHO). Thus furan reacts with α,β-unsatd. carbonyl compds. not only according to the diene synthesis (cf. Diels, *et al.*, *C. A.* **20**, 430-6, 3951), but under certain conditions it gives condensation products without cyclization, similar to pyrrole (Diels, *loc. cit.*), by combining in the α-position with 1 and 2 acrolein mols. with the transposition of its atom or atoms to the double bond of acrolein. This reaction does not take place in the absence of SO_2 and in the presence of acidic compds., such as org. and inorg. acids, H_2S, etc., and proceeds satisfactorily only at the optimum SO_2 concn. SO_2 is sp. in its action and does not catalyze the condensation of furan with some other similar unsatd. aldehydes and acids. Thus acrylic acid with furan gave only the polymerized acid. Hydroquinone is added to stabilize acrolein, since without it the entire aldehyde becomes polymerized. A mixt. of 146 g. furan (redistd. over Na), 120 g. of dry acrolein contg. 0.5 g. hydroquinone and 0.5 ml. of aq. SO_2 (approx. 10 mg. SO_2) in a glass-lined autoclave was heated at 100° for 1 hr. and then vacuum distd., giving 17 g. I and 30.4 g. II. I, b_{11} 81°, d_4^{19} 1.0591, d_4^{20} 1.0574, n_D^{20} 1.4772, M. R. 33.16 (calcd. 33.05), mol. wt. 124. The *acid* (III), b_{10} 135° (slight decompn.), m. 50°, prepd. by oxidizing I in H_2O with $AgNO_3$, converting the acid into the Na salt with NaOH and decomps. it in the filtrate with 20% H_2SO_4. *Me ester*, b_{13} 89°, d_4^{20} 1.088, n_D^{20} 1.4662, M. R. 39.23, prepd. with CH_2N_2 in Et_2O. *β-(α-Tetrahydrofuryl)propionic acid*, b_{11} 137°, d_4^{19} 1.1272, n_D^{19} 1.402, M. R. 35.12, prepd. by hydrogenating 10.5 g. III in 5% NaOH (150 ml.) in the presence of 5 ml. of 1% PdCl, and 100 mg. gum arabic. II, m. 41.2° (petr. ether), mol. wt. 177.14 (calcd. 180); the *dioxime*, m. 132-3°. The *acid*, m. 154-4.5° (H_2O), gave with CH_2N_2 the *di-Me ester*, b_{12} 172-4°, m. 50.5-7.5°, and on boiling in concd. HCl *biterulinic acid*, m. 156.5-7.5°. Aprox. 35 references. II. **Condensation of tetra- and hexahydrobenzaldehyde with acrolein.** A. Ya. Berlin and S. M. Sherlin. *Ibid.* 16-21.—The condensation of Δ^3-tetrahydrobenzaldehyde (I) and hexahydrobenzaldehyde (II) with acrolein in the presence of SO_2 and hydroquinone as described above gave Δ^3-*cyclohexene-1-formyl-1-β-propionaldehyde* (III) and *cyclohexane-1-formyl-1-β-propionaldehyde* (IV), resp. Thus, the condensation of I and II and that of furan with acrolein proceeds analogously. I and II were obtained by the method of Diels and Alder (*C. A.* **22**, 1144). The trimer of I is formed in a few hrs. on adding a drop of HCl or H_2SO_4; it m. 176°. A mixt. of 17.8 g. II, 20 ml. of dry acrolein stabilized with hydroquinone and 4 mg. SO_2 when heated in sealed tubes at 100-5° for 3 hrs. and then vacuum-distd. gave 6.2 g. IV, b_7 120-1°, b_{11} 132-3°. It quickly reduces Fehling soln. and $AgNO_3$ in NH_4OH and is polymerized to a heavy mass, which on heating is depolymerized to IV, mol. wt. 176, 184 (calcd. for $C_{11}H_{16}O_2$, 168); α could not be detd. because of the rapid polymerization of IV. The *acid*, m. 130-1°. The *lactone*, b_5 110-12°, d_4^{19} 1.08, n_D^{19} 1.483, M. R. 45.21. The condensation of 34 g. I with 35 ml. acrolein as above gave 14 g. of a product, b_5 129-35°. As in the case of IV, it was impossible to obtain pure III because of the rapid polymerization and the incomplete depolymerization on heating. III, b_{11} 140-2°, b_{13} 146-8°. The *acid*, m. 161°. When its Na salt was hydrogenated

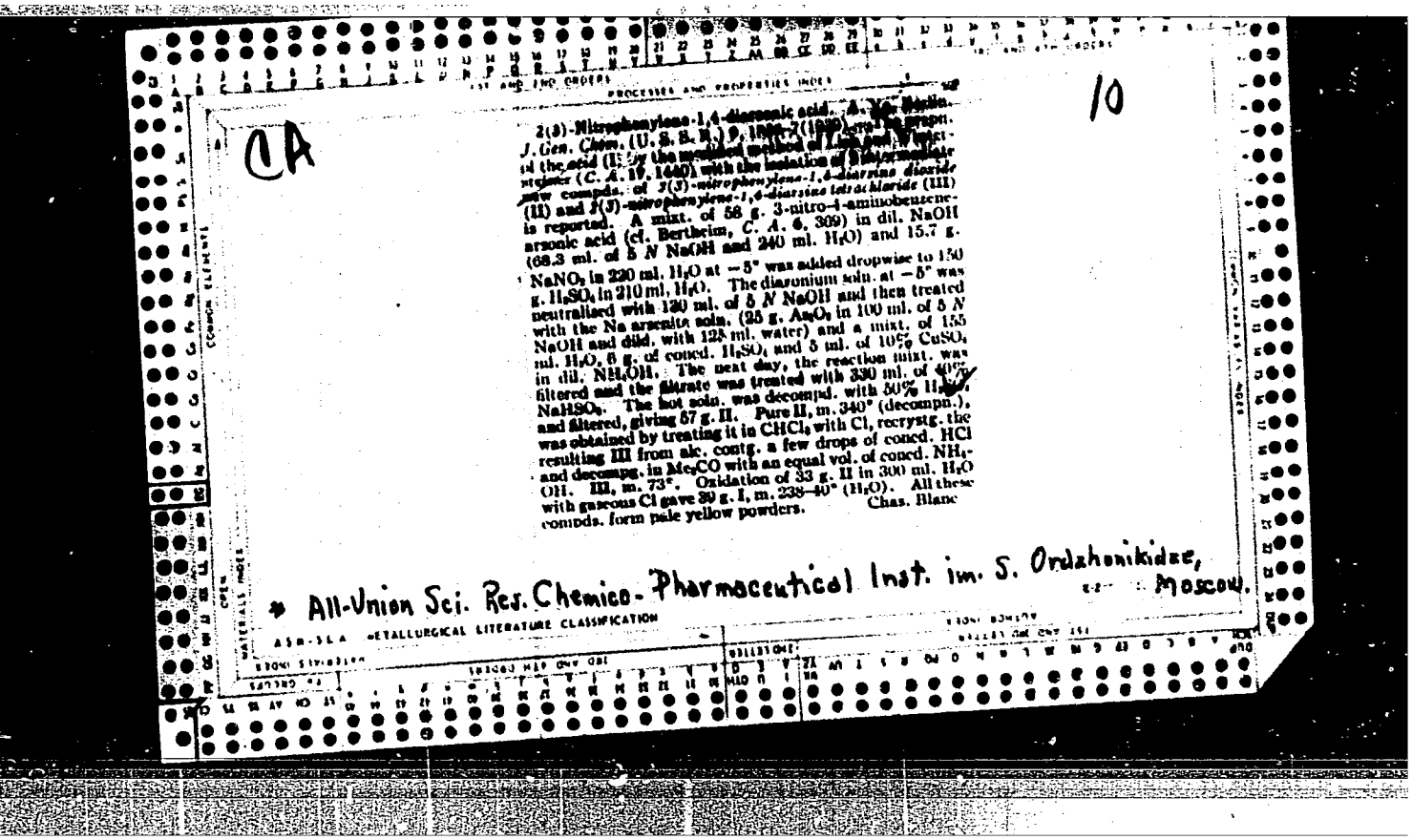


in the presence of colloidal Pd it gave the IV acid. III. Polymerization of acrolein and acrylic acid and the structure of their dimers. S. M. Sherlin, A. Ya. Berlin, T. A. Serebrennikova and F. E. Rabinovich. *Ibid.* 22 34.—It has been shown above that when acrolein (I) and acrylic acid (II) are heated with furan in the absence of the SO_2 catalyst they are easily polymerized to corresponding dimers and polymers. It was of interest to investigate the structure and the mechanism of formation of I and II dimers. Autoclaving 100 ml. of dry I in 100 ml. C_6H_6 in the presence of a little hydroquinone at 170° for 5 hrs. gave 26 g. of I dimer, which proved to be *3-formyl-2,3-dihydropyran*, $O.CH_2.CH(CHO).CH_2.CH:CH$, (III), b. 146, b_{30} 40 0.5°, d_{20}^{20} 1.0889, d_4^{20} 1.0795, n_D^{20} 1.466, M. R. 28.73 (calcd. for $C_6H_8O_2$, 28.89), mol. wt. 112. The same results were obtained with furan and Et_2O as solvents. Without the stabilization of I with hydroquinone only high-mol. polymers and no dimer were formed. III on standing for a few days forms a trimer (I hexamer), a stable viscous mass. By analogy with the Alder interpretation of the condensation of cyclopentadiene to dimer and polymer by the "diene synthesis" (C. *A.* 25, 1806), the formation of dimer, trimer and polymers of I can be schematically represented as a chain of condensed pyran nuclei, thus:

III gives only a monourea derivative, m. 123°, and a monoxime, b_{15} 100°, b_{30} 101 2°, which in the catalytic hydrogenation absorb only 1 mol. H, giving *3-formyltetrahydropyran semicarbazone*, m. 134°, and *oxime*, b_{15} 102 4°, resp. The oxime on boiling in Ac_2O gave the *nitrile*, b_{30} 77°, d_4^{20} 1.0171, n_D^{20} 1.4428, M. R. 28.92. This in alk. soln. + pet. ether with HCl gas gave *3,4-tetrahydropyran-3-carboxylate*, b_{30} 101 3°, d_4^{20} 1.0410, n_D^{20} 1.453, M. R. 40.21 (calcd. 40.24). III (11.5 g.) in 30 ml. Et_2O with PhMgBr (from 5 g. Mg and 35 g. PhBr in 120 ml. Et_2O) gave 12 g. (*2,3-dihydro-3-pyranyl*)*phenylcarbinol*, $O.CH_2.CH[CH(OH)Ph].CH_2.CH:CH$, b_{30} 166°, d_4^{20} 1.117, n_D^{20} 1.5405, M. R. 54.15. III oxidized with $AgNO_3$ in alk. soln. gave the *acid* (Na salt). Heating 9 g. II in 9 ml. furan in a sealed tube at 160° for 5 hrs. gave 3 g. of II dimer, which proved to be *acrylhydroacrylic acid*, $CH_2:CHCO_2CH_2CH_2CO_2H$ (IV), b_{30} 146-8°, b_{10} 130°, d_4^{20} 1.1088, n_D^{20} 1.4522, M. R. 64.78 (calcd. for $C_6H_8O_4$, 65.03), mol. wt. 296 (calcd. 144). Thus, the mol. wt. of IV, detd. in C_6H_6 by the cryoscopic method, is nearly equal to the fourfold value for II. This is ascribed to the assocn. process characteristic for many carboxylic acids (cf. Vorlander, *Ann.* 294, 257 (1897)). The following reactions proved conclusively that IV is a dimer of II. IV absorbs only 1 H, forming *propionylhydroacrylic acid*, $EtCO_2CH_2CH_2CO_2H$, b_{10} 145-6°. The *Me ester*, b_{10} 85°, d_4^{20} 1.0715, d_4^{25} 1.0720, n_D^{20} 1.4204, M. R. 37.82 (calcd. for $C_7H_{12}O_4$, 37.83), mol. wt. 148 (calcd. 160). This proved to be identical with the Me ester of the same acid synthesized from EtCOCl with Me hydroacrylate. While the polymerization of I in furan, C_6H_6 and Et_2O proceeds analogously, the condensation of II is influenced by the solvents. Thus, II in furan gave only the dimer and in C_6H_6 higher polymers with only traces of dimer.

Chas. Blanc





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2(3)-Nitrophenylene-1,4-diarsonic acid. A. [illegible]. J. Gen. Chem. (U. S. S. R.) 9, 1899–7(1939).—The prepn. of the acid (I) by the modified method of Lesser and Weingarten (C. A. 19, 1440) with the isolation of [illegible] new compds. of 2(3)-nitrophenylene-1,4-diarsine dioxide (II) and 2(3)-nitrophenylene-1,4-diarsine tetrachloride (III) is reported. A mixt. of 58 g. 3-nitro-4-aminobenzenearsonic acid (cf. Bertheim, C. A. 6, 309) in dil. NaOH (68.3 ml. of 5 N NaOH and 240 ml. H_2O) and 15.7 g. $NaNO_2$ in 220 ml. H_2O at −5° was added dropwise to 150 g. H_2SO_4 in 210 ml. H_2O. The diazonium soln. at −5° was neutralized with 120 ml. of 5 N NaOH and then treated with the Na arsenite soln. (25 g. As_2O_3 in 100 ml. of 5 N NaOH and dild. with 128 ml. water) and a mixt. of 155 ml. H_2O, 6 g. of concd. H_2SO_4 and 5 ml. of 10% $CuSO_4$ in dil. NH_4OH. The next day, the reaction mixt. was filtered and the filtrate was treated with 330 ml. of 40% $NaHSO_3$. The hot soln. was decompd. with 50% H_2SO_4 and filtered, giving 57 g. II. Pure II, m. 340° (decompn.), was obtained by treating it in $CHCl_3$ with Cl, recrystg. the resulting III from alc. contg. a few drops of concd. HCl and decompg. in Me_2CO with an equal vol. of concd. NH_4OH. III, m. 73°. Oxidation of 33 g. II in 300 ml. H_2O with gaseous Cl gave 39 g. I, m. 238–40° (H_2O). All these compds. form pale yellow powders. Chas. Blanc

* All-Union Sci. Res. Chemico-Pharmaceutical Inst. im. S. Ordzhonikidze, Moscow.

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

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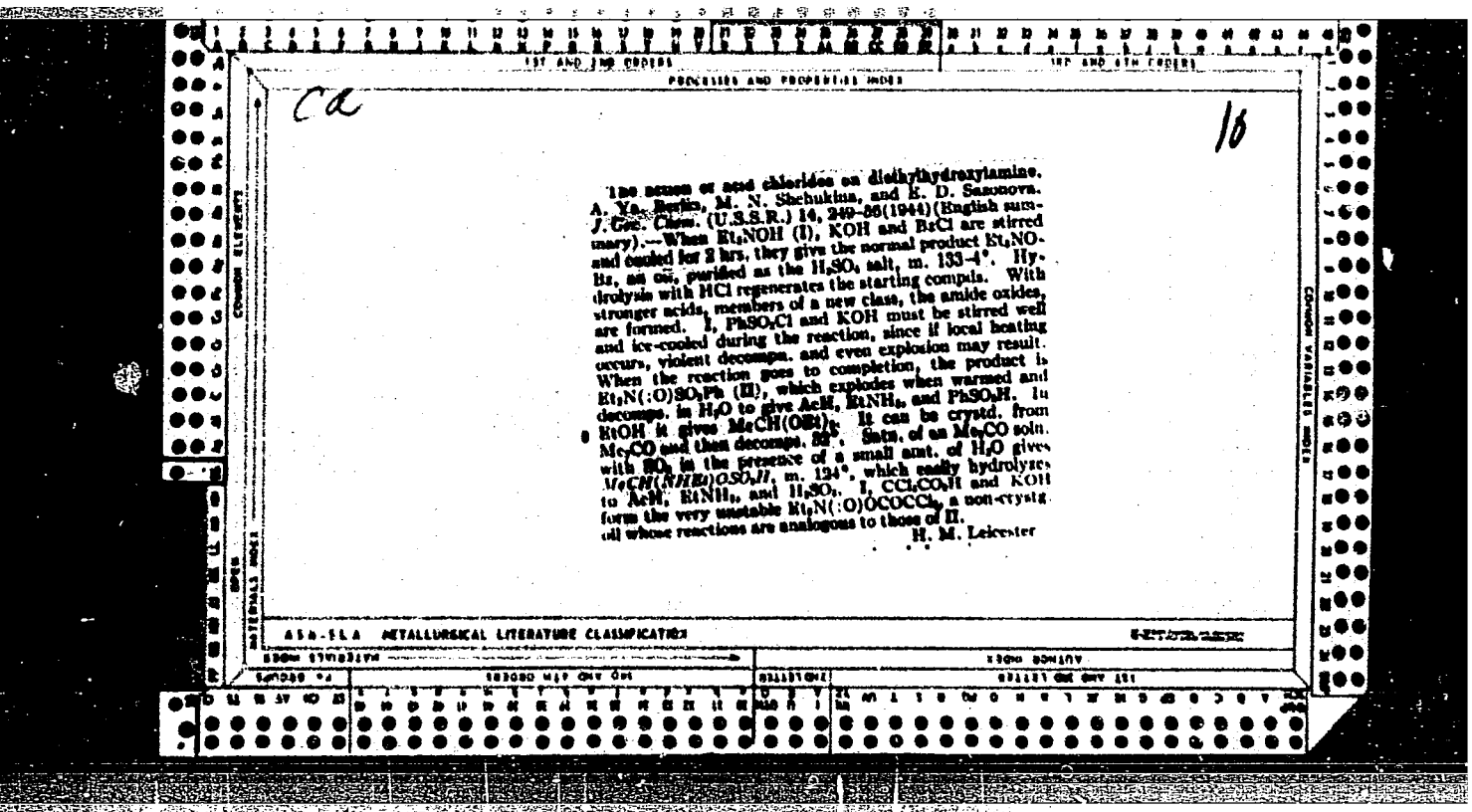
Condensation and polymerization of α,β-unsatd. aldehydes and acids. IV. Condensation of α-methyleneglutaric acid anhydride with butadiene. A. Ya. Berlin. *J. Gen. Chem.* (U. S. S. R.) 14, 110-19(1944); cf. *C. A.* 32, 5386[9].—α-Methyleneglutaric acid (5.5 g.) and 30 cc. AcCl were warmed on a water bath for 3 hrs. to yield 1.95 g. *α-methyleneglutaric anhydride*, b_{8} 120-9°, m. 51° (from abs. Et_2O). The above (2.75 g.) and 3.8 g. butadiene in 5 cc. dry benzene were heated in a sealed tube for 2 hrs. at 110°, the mixt. treated with warm 5% NaOH, the soln. acidified and extd. with Et_2O to yield *1-carboxy-3-cyclohexene-1-propionic acid*, m. 100-1° (from water). G. M. K.

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

MATERIALS INDEX

AUTHOR INDEX



Ca

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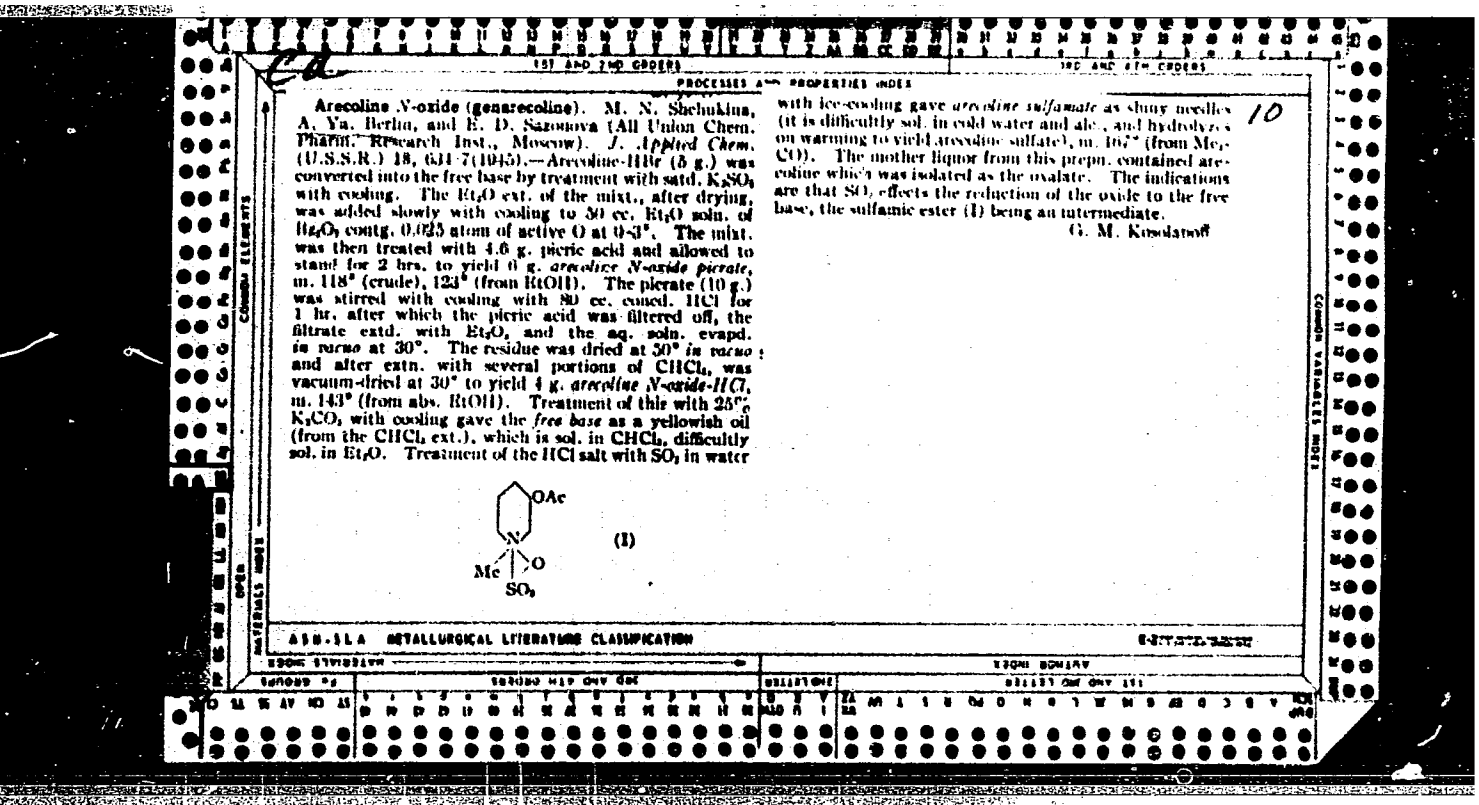
The action of acid chlorides on diethylhydroxylamine. A. Ya. Berlin, M. N. Shchukina, and E. D. Saxonova. *J. Gen. Chem.* (U.S.S.R.) 14, 249–56(1944)(English summary).—When Et_2NOH (I), KOH and BzCl are stirred and cooled for 2 hrs., they give the normal product Et_2NOBz, an oil, purified as the H_2SO_4 salt, m. 133–4°. Hydrolysis with HCl regenerates the starting compds. With stronger acids, members of a new class, the amide oxides, are formed. I, $PhSO_2Cl$ and KOH must be stirred well and ice-cooled during the reaction, since if local heating occurs, violent decompn. and even explosion may result. When the reaction goes to completion, the product is $Et_2N(:O)SO_2Ph$ (II), which explodes when warmed and decomps. in H_2O to give AcH, $EtNH_2$, and $PhSO_2H$. In EtOH it gives $MeCH(OEt)_2$. It can be crystd. from Me_2CO and then decomps. 88°. Satn. of an Me_2CO soln. with SO_2 in the presence of a small amt. of H_2O gives $MeCH(NHEt)OSO_2H$, m. 124°, which easily hydrolyzes to AcH, $EtNH_2$, and H_2SO_4. I, CCl_3CO_2H and KOH form the very unstable $Et_2N(:O)OCOCCl_3$, a non-crystg. oil whose reactions are analogous to those of II.

H. M. Leicester



1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

Arecoline *N*-oxide (gensarecoline). M. N. Shchukina, A. Ya. Berlin, and E. D. Sazonova (All Union Chem. Pharm. Research Inst., Moscow). *J. Applied Chem. (U.S.S.R.)* **18**, 631-7(1945).—Arecoline-HBr (5 g.) was converted into the free base by treatment with satd. K_2SO_3 with cooling. The Et_2O ext. of the mixt., after drying, was added slowly with cooling to 30 cc. Et_2O soln. of Hg_2O_2 contg. 0.025 atom of active O at 0-3°. The mixt. was then treated with 4.6 g. picric acid and allowed to stand for 2 hrs. to yield 6 g. *arecoline N-oxide picrate*, m. 118° (crude), 123° (from EtOH). The picrate (10 g.) was stirred with cooling with 80 cc. concd. HCl for 1 hr. after which the picric acid was filtered off, the filtrate extd. with Et_2O, and the aq. soln. evapd. *in vacuo* at 30°. The residue was dried at 50° *in vacuo* and after extn. with several portions of $CHCl_3$, was vacuum-dried at 30° to yield 4 g. *arecoline N-oxide-HCl*, m. 143° (from abs. EtOH). Treatment of this with 25% K_2CO_3 with cooling gave the *free base* as a yellowish oil (from the $CHCl_3$ ext.), which is sol. in $CHCl_3$, difficultly sol. in Et_2O. Treatment of the HCl salt with SO_3 in water with ice-cooling gave *arecoline sulfamate* as shiny needles (it is difficultly sol. in cold water and alc., and hydrolyzes on warming to yield arecoline sulfate), m. 107° (from Me_2CO). The mother liquor from this prepn. contained arecoline which was isolated as the oxalate. The indications are that SO_3 effects the reduction of the oxide to the free base, the sulfamic ester (I) being an intermediate.

G. M. Kosolapoff

(I)

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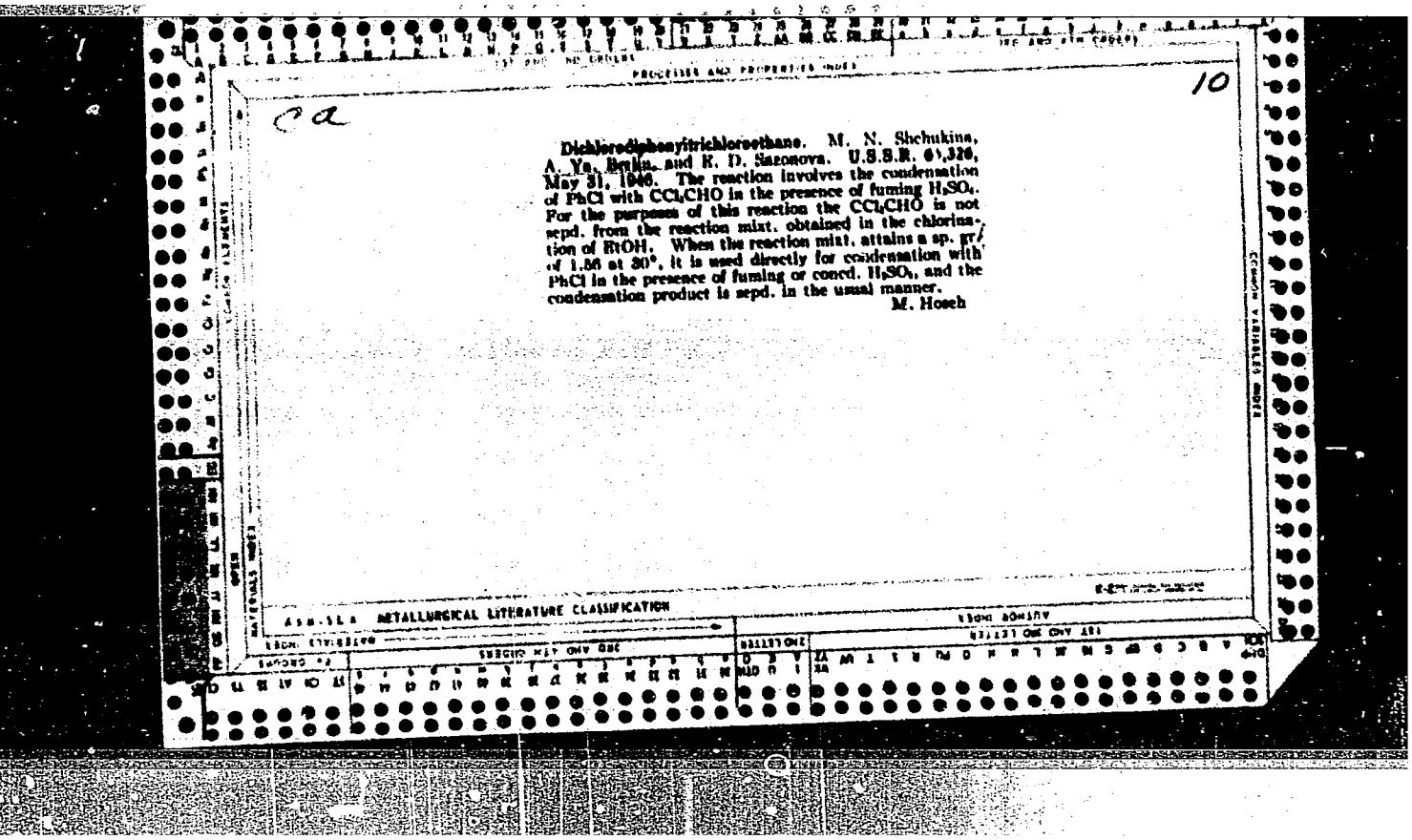
A59-51A METALLURGICAL LITERATURE CLASSIFICATION



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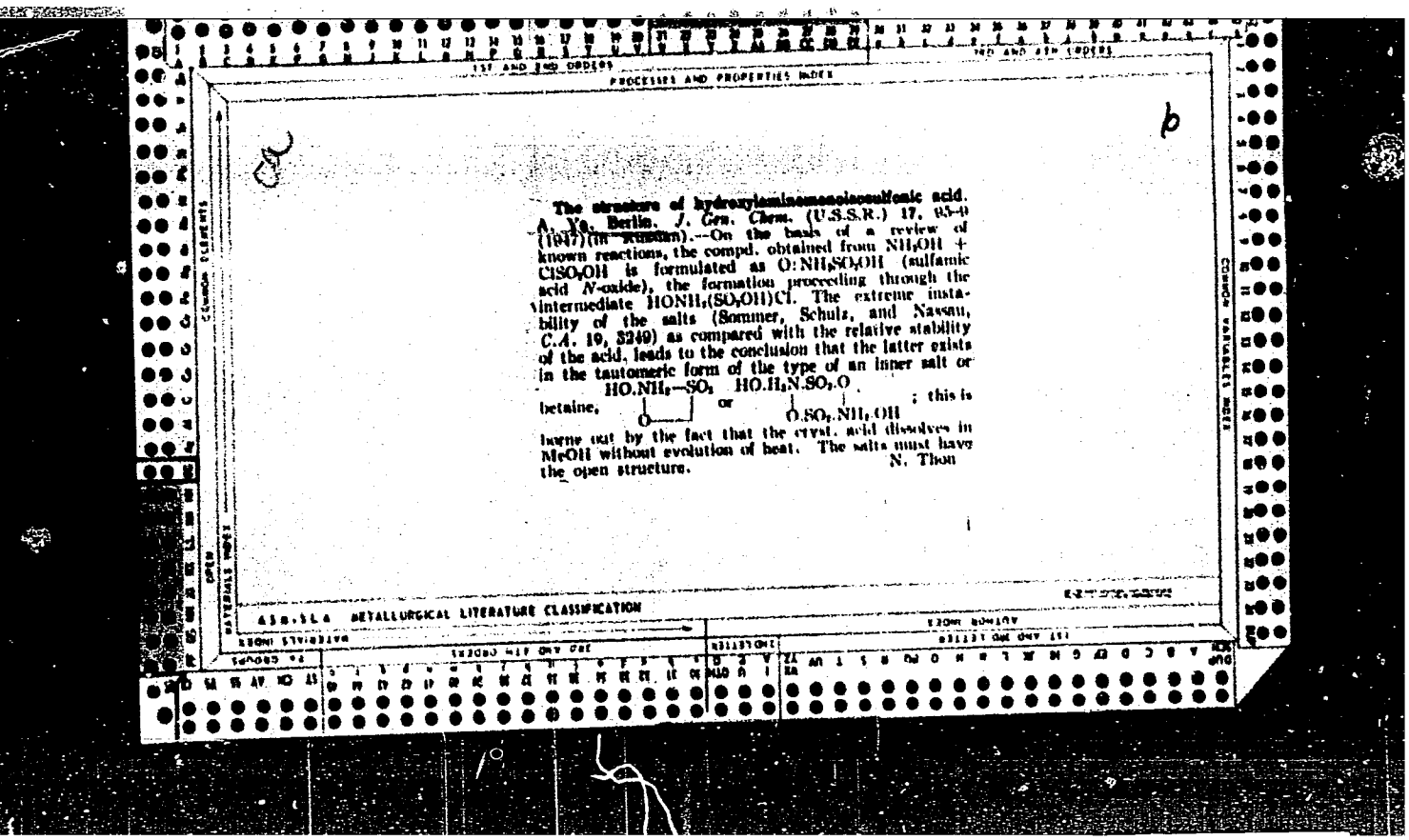
Dichlorodiphenyltrichloroethane. M. N. Shchukina, A. Ya. Berlin, and K. D. Sazonova. U.S.S.R. 61,326, May 31, 1946. The reaction involves the condensation of PhCl with CCl_3CHO in the presence of fuming H_2SO_4. For the purposes of this reaction the CCl_3CHO is not sepd. from the reaction mixt. obtained in the chlorination of EtOH. When the reaction mixt. attains a sp. gr. of 1.56 at 30°, it is used directly for condensation with PhCl in the presence of fuming or concd. H_2SO_4, and the condensation product is sepd. in the usual manner.

M. Hoseh



The structure of hydroxylaminomonosulfonic acid. A. Yu. Berlin. *J. Gen. Chem.* (U.S.S.R.) 17, 85-9 (1947) (in Russian).--On the basis of a review of known reactions, the compd. obtained from NH_2OH + $ClSO_3OH$ is formulated as $O:NH_2SO_3OH$ (sulfamic acid *N*-oxide), the formation proceeding through the intermediate $HONH_2(SO_3OH)Cl$. The extreme instability of the salts (Sommer, Schulz, and Nassau, *C.A.* 19, 3249) as compared with the relative stability of the acid, leads to the conclusion that the latter exists in the tautomeric form of the type of an inner salt or betaine, $HO.NH_2{-}SO_3$ or $HO.H_2N.SO_2.O$; this is borne out by the fact that the cryst. acid dissolves in MeOH without evolution of heat. The salts must have the open structure.

N. Thon

BERLIN, A. YA. PA 30/49T15

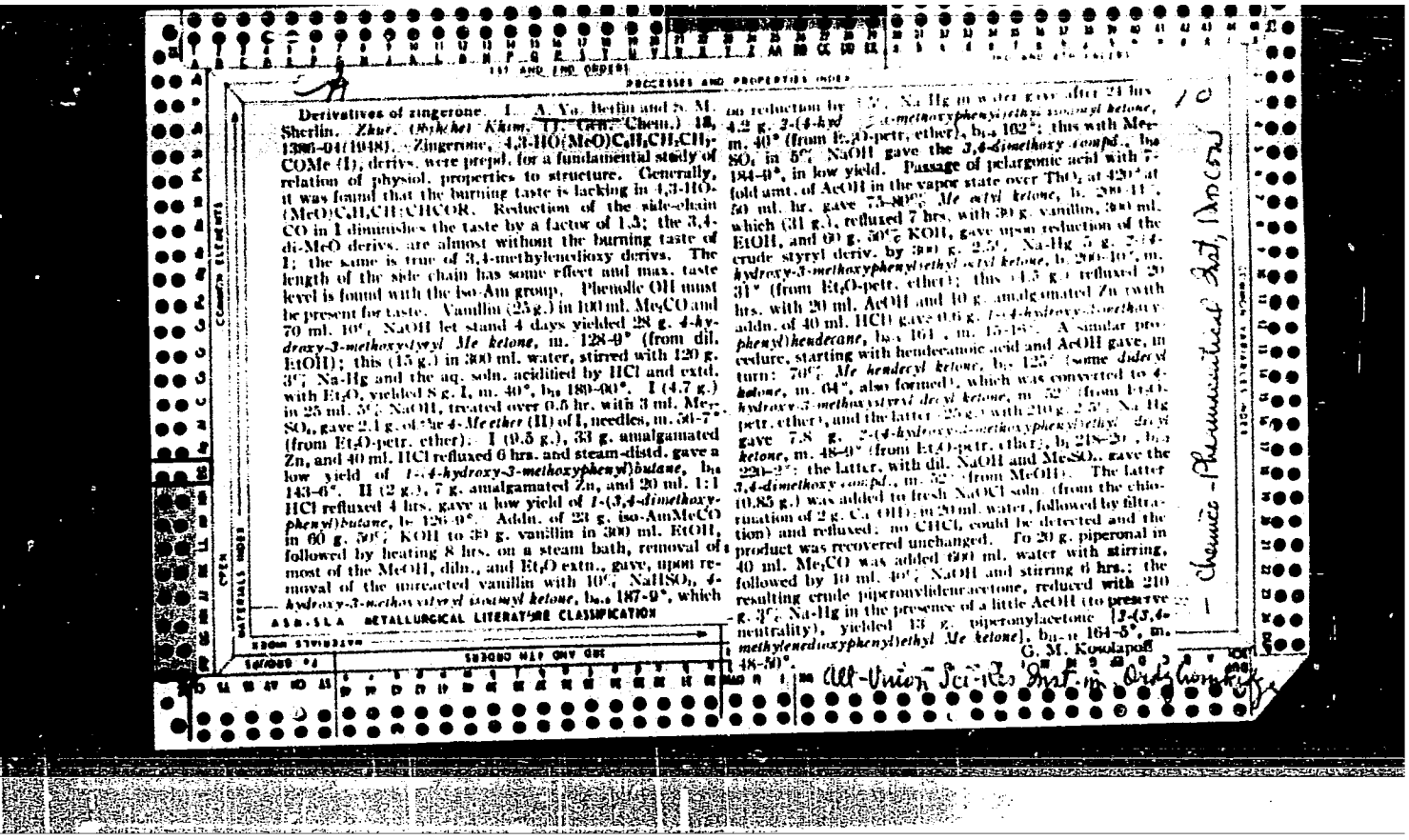
USSR/Chemistry - Synthesis Sep 48
Chemistry - Sulfone, n-Aminophenyl-Chlormethyl

"N-Aminophenyl-Chlormethylsulfone," A. Ya. Berlin, All-Union Chem Phar Sci Res Inst imeni S. Ordzhonikidze, Moscow, 2 pp

"Zhur Obshch Khimii" Vol XVIII, No 9

Describes synthesis of n-acetylaminophenyl-chlormethylsulfone and n-aminophenyl-chlormethylsulfone. Submitted 16 May 47.

30/49T15



Derivatives of zingerone. I. A. Yu. Berlin and S. M. Sherlin. *Zhur. Obshchei Khim.*, J. Gen. Chem.) 18, 1386–94(1948). —Zingerone, 4,3-HO(MeO)C$_6$H$_3$CH$_2$CH$_2$COMe (I), derivs. were prepd. for a fundamental study of relation of physiol. properties to structure. Generally, it was found that the burning taste is lacking in 4,3-HO(MeO)C$_6$H$_3$CH:CHCOR. Reduction of the side-chain CO in I diminishes the taste by a factor of 1.5; the 3,4-di-MeO derivs. are almost without the burning taste of I; the same is true of 3,4-methylenedioxy derivs. The length of the side chain has some effect and max. taste level is found with the iso-Am group. Phenolic OH must be present for taste. Vanillin (25 g.) in 100 ml. MeCOEt and 70 ml. 10% NaOH let stand 4 days yielded 28 g. *4-hydroxy-3-methoxystyryl Me ketone*, m. 128–9° (from dil. EtOH); this (15 g.) in 300 ml. water, stirred with 120 g. 3% Na-Hg and the aq. soln. acidified by HCl and extd. with Et$_2$O, yielded 8 g. I, m. 40°, b$_6$ 180–90°. I (4.7 g.) in 25 ml. 5% NaOH, treated over 0.5 hr. with 3 ml. Me$_2$SO$_4$, gave 2.1 g. of the *4-Me ether* (II) of I, needles, m. 50–7° (from Et$_2$O-petr. ether). I (9.5 g.), 33 g. amalgamated Zn, and 40 ml. HCl refluxed 6 hrs. and steam-distd. gave a low yield of *1-(4-hydroxy-3-methoxyphenyl)butane*, b$_6$ 143–6°. II (2 g.), 7 g. amalgamated Zn, and 20 ml. 1:1 HCl refluxed 4 hrs. gave a low yield of *1-(3,4-dimethoxyphenyl)butane*, b$_5$ 126–9°. Addn. of 23 g. iso-AmMeCO in 60 g. 50% KOH to 30 g. vanillin in 300 ml. EtOH, followed by heating 8 hrs. on a steam bath, removal of most of the MeOH, dilu., and Et$_2$O extn., gave, upon removal of the unreacted vanillin with 10% NaHSO$_3$, *4-hydroxy-3-methoxystyryl isoamyl ketone*, b$_{0.6}$ 187–9°, which on reduction by 3% Na-Hg in water gave after 24 hrs. 4.2 g. *3-(4-hydroxy-3-methoxyphenyl)ethyl isoamyl ketone*, m. 40° (from Et$_2$O-petr. ether), b$_{0.6}$ 162°; this with Me$_2$SO$_4$ in 5% NaOH gave the *3,4-dimethoxy compd.*, b$_6$ 184–9°, in low yield. Passage of pelargonic acid with 7-fold amt. of AcOH in the vapor state over ThO$_2$ at 420° at 50 ml. hr. gave 73–80% *Me octyl ketone*, b$_{15}$ 200–11°, which (31 g.), refluxed 7 hrs. with 30 g. vanillin, 300 ml. EtOH, and 90 g. 50% KOH, gave upon reduction of the crude styryl deriv. by 3% g. 2.5% Na-Hg 5 g. *3,4-hydroxy-3-methoxyphenyl)ethyl octyl ketone*, b$_{0.5}$ 200–10°, m. 31° (from Et$_2$O-petr. ether); this (4.5 g.) refluxed 20 hrs. with 20 ml. AcOH and 10 g. amalgamated Zn (with addn. of 40 ml. HCl gave 0.6 g. *1-(4-hydroxy-3-methoxyphenyl)hendecane*, b$_{0.5}$ 161°, m. 15–16°. A similar procedure, starting with hendecanoic acid and AcOH gave, in turn: 70% *Me hendecyl ketone*, b$_5$ 125° (some *didecyl ketone*, m. 64°, also formed), which was converted to *4-hydroxy-3-methoxystyryl decyl ketone*, m. 52° (from 1:1 petr. ether), and the latter (25 g.) with 200 g. 2.5% Na-Hg gave 7.8 g. *3-(4-hydroxy-3-methoxyphenyl)ethyl decyl ketone*, m. 48–9° (from Et$_2$O-petr. ether), b$_1$ 218–20°, b$_{0.5}$ 221–2°; the latter, with dil. NaOH and Me$_2$SO$_4$, gave the *3,4-dimethoxy compd.*, m. 32° (from MeOH). The latter (0.85 g.) was added to fresh NaOCl soln. (from the chlorination of 2 g. Ca(OH)$_2$ in 20 ml. water, followed by filtration) and refluxed; no CHCl$_3$ could be detected and the product was recovered unchanged. To 20 g. piperonal in 40 ml. Me$_2$CO was added 200 ml. water with stirring, followed by 10 ml. 40% NaOH and stirring 6 hrs.; the resulting crude piperonylideneacetone, reduced with 210 g. 3% Na-Hg in the presence of a little AcOH (to preserve neutrality), yielded 13 g. piperonylacetone [*3-(3,4-methylenedioxyphenyl)ethyl Me ketone*], b$_{0.4-0.6}$ 164–5°, m. 48–50°.

G. M. Kosolapoff

All-Union Sci-Res Inst. of Org. Synthesis

BERLIN, A. Ia.

A. Ia. Berlin, n-Amino-phenyl-chloro-methyl-sulfone. p. 1716

n-acetyl-amino-phenyl-chloro-methyl-sulfone and n-amino-phenyl-chloro-methyl-sulfone were synthesized.

The Ordzhonikidze All-Union Chemico-Pharmaceutical Scientific Research Institute, Moscow. May 16, 1947

SO: Journal of General Chemistry (USSR) 28, (80) No. 9 (1948)

BERLIN, A. IA.

A. Ia. Berlin and In. V. Markova, Fatty aromatic γ-derivatives of aceto acetic ester. p. 1791

A series of β-keto ester derivatives of acetoacetic ester were synthesized and their properties studied.

The Orzhonikidze, All Union Scientific Research Inst. of Pharmaceutical Chemistry Moscow, September 29, 1947

SO: Journal of General Chemistry (USSR) 28, (80) No. 10 (1948):

BERLIN, A. YA

PA76T11

USSR/Chemistry - Heterocyclics Jun 1948
Chemistry - Organic Compounds

"Transformation of Heterocyclics: 2-Phenylthiazol-4-Carbonic Acid From 2-Phenyl-4-(Carbethoxymethylamino-methyleme)-Oxazolin-5-On," A. Ya. Berlin, V. I. Maynind, All-Union Sci Res Chemicophar Inst imeni S. Ordzhonikidze, 3 pp

"Dok Ak Nauk SSSR" Vol LX, No 7 - pp. 1181-3

Describes and analyzes experiment involving subject reaction. Submitted Mar 1948.

76T11

BERLIN, A. YA.

27608

BERLIN, A. YA. I MARKOVA, YU. V. Proizvodnyye Tsingerona. (Soobsh.) 4. Zhurnal obshchey Khimii, 1949, Vyp. 8, s. 1567-70.

SO: Letopis' Zhurnal'nykh Statey, Vol. 37, 1949

BERLIN, A. Ya.

PA 65/49T22

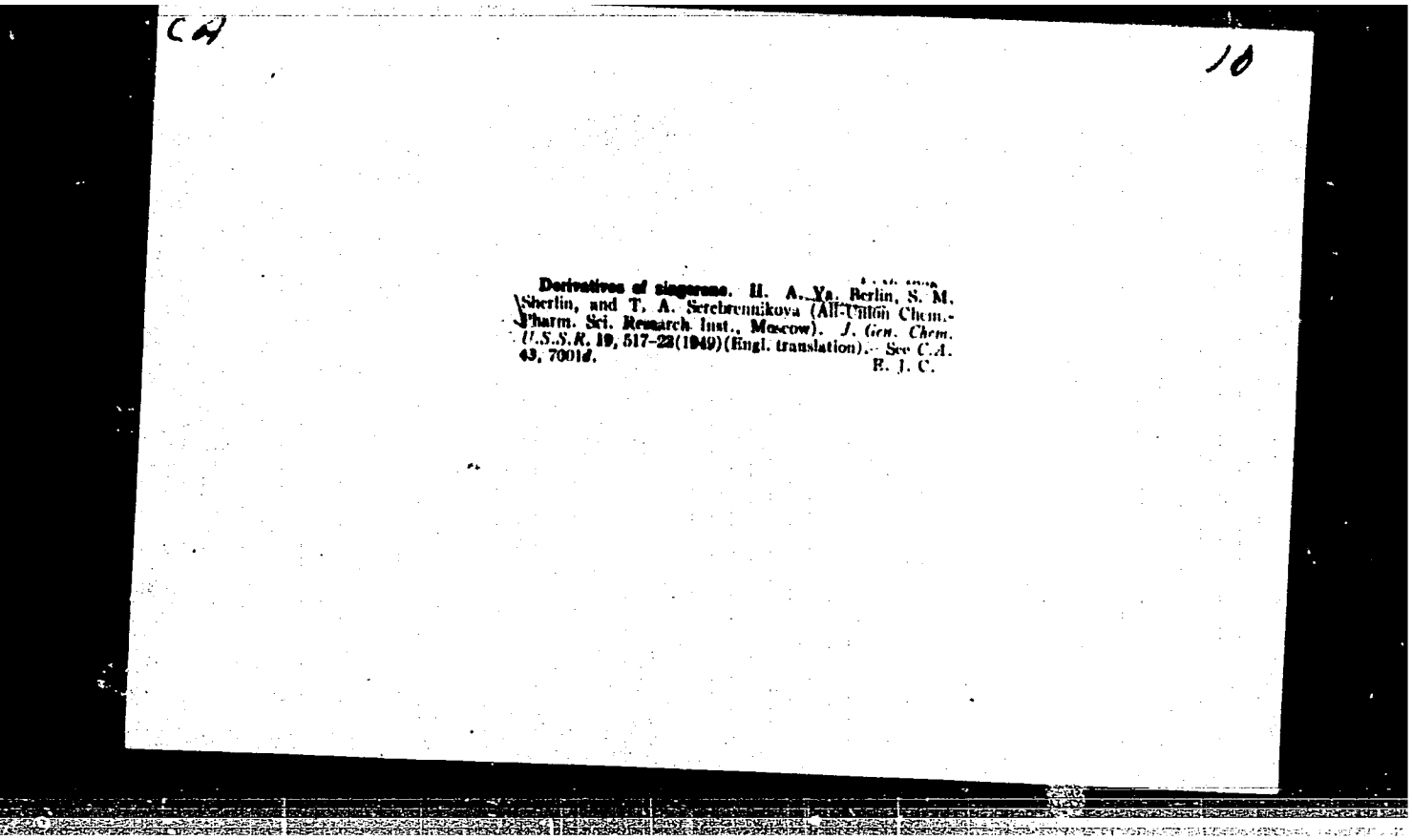
USSR/Chemistry - Chloral Apr 49
Anilides

"The Condensation of Acylanilides With Chloral," A. Ya. Berlin, M. N. Shchukina, Ye. D. Sazonova, All-Union Sci Res Chemicopharm Inst imeni S. Ordzhonikidze, 6 pp

"Zhur Obshch Khim" Vol XIX, No 4

Study of subject reaction in the presence of H_2SO_4 established that acetanilide and phthalanil enter into the reaction, while succinanil, in the observed experiments, did not. Gives products of the reaction of acetanilide and the phthalanil with the chloral.

65/49T22



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Derivatives of sinigrane. II. A. Ya. Berlin, S. M. Sherlin, and T. A. Serebrennikova (All-Union Chem.-Pharm. Sci. Research Inst., Moscow). *J. Gen. Chem. U.S.S.R.* **19**, 517–23(1949)(Engl. translation).—See *C.A.* **43**, 7001*d*.
R. J. C.

BERLIN, A. Ya.

PA 65/49T32

USSR/Chemistry - Ringerome Apr 49
Organic Compounds

"Ringerome Derivatives, III," A. Ya. Berlin, S. M. Sherlin (deceased), T. A. Serebrennikova, All-Union Sci Res Chemicopher Inst imeni S. Ordzhonikidze, Moscow, 9½ pp

"Zhur Obshch Khim" Vol XIX, No 4

Synthesized a series of these compounds, characterized by the length of the alkoxyl groups, by the presence of an amine group in the aromatic nucleus in place of a phenol hydroxyl, and by a change in the position of the carboxyl group in the side chain. Discovered that the approach of the carboxyl group to the amine nucleus in the [illegible] of the side chain did not result in a decolorization of the caustic tests of these compounds. Submitted 12 Sep 47.

[redacted]

6/49T32

BERLIN, A. YA.

PA 64/49T25

Jun 49
Chemistry - Synthesis

"4-Oxy-3-methoxypiperidine," A. Ya. Berlin, All-Union Sci Res Chemicopharm Inst imeni S. Ordzhonikidze, Moscow, 2 pp

"Zhur Obshch Khim" Vol XIX, No 6

Synthesized this substance, and determined more precise constants for β-methoxy- γ -pyrone. Submitted 7 Mar 48.

64/49T25

BERLIN, A. YA.

PA 149T38

USSR/Chemistry - Zingerone Synthesis

Aug 49

"Zingerone Derivatives, IV," A. Ya. Berlin, Yu. V. Markova, All-Union Sci Res Chemicopharm Inst imeni Ordzhonikidze, Moscow, 3½ pp

"Zhur Obshch Khim" Vol XIX, No 8

[illegible] synthesized series of ketones [illegible] CH_3O) $C_6H_3CH_2CH_2$ [illegible] $CH_2)_nC_6H_5$ (with n equal to 1, 2, 3, and 4) by [illegible] of benzylmethylcarbinol chloride with ap- [illegible] [illegible] derivatives of an [illegible] [illegible] obtained by synthesis and [illegible] Submitted 13 Mar 48.

CA

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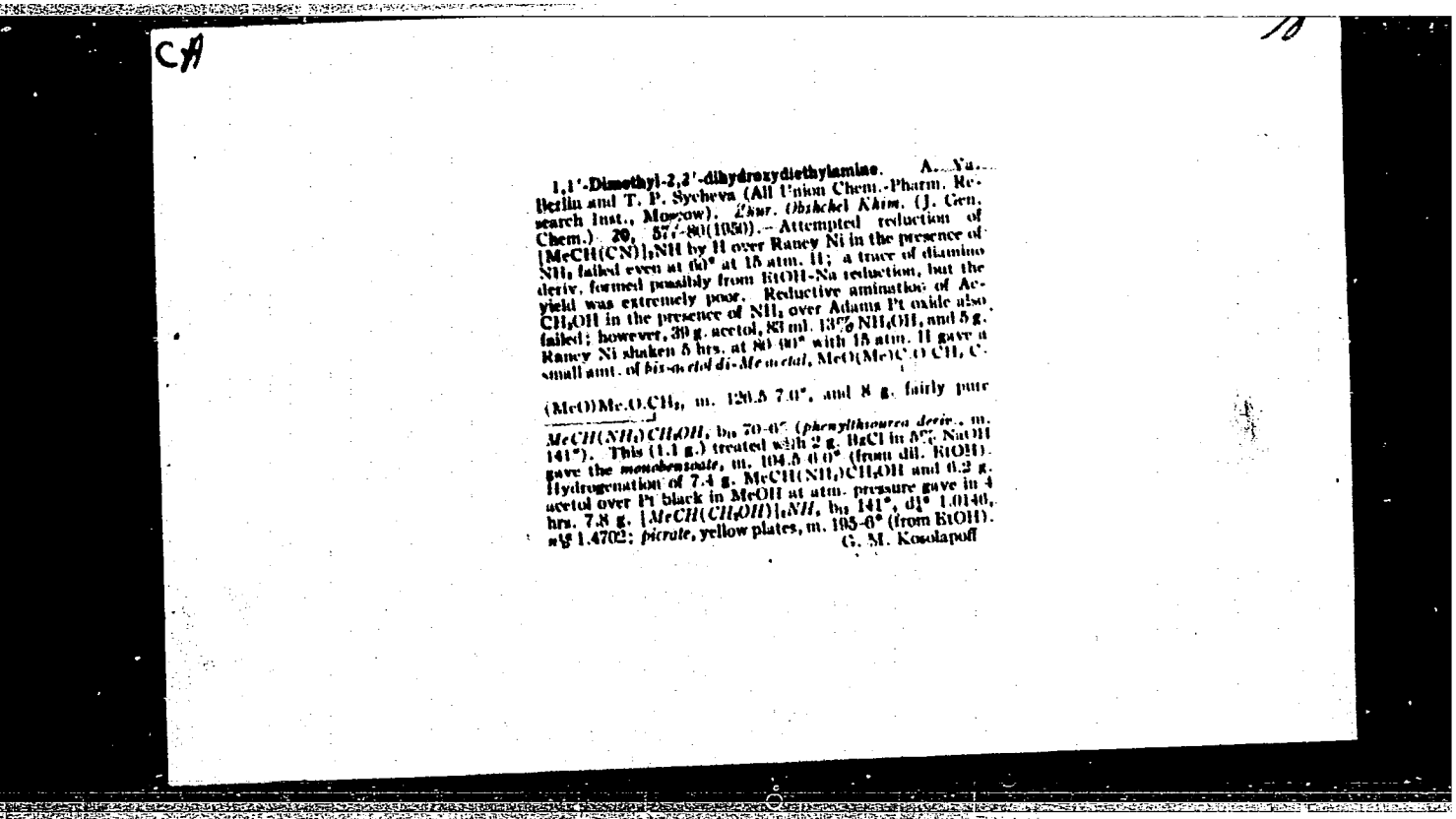
Condensation of acylanilides with chloral. A. Ya. Berlin, M. N. Shchukina, and E. D. Sazonova (All-Union Sci. Research Chem.-Pharm. Inst., Moscow). *J. Gen. Chem.* (U.S.S.R.) 19, 639-45(1949)(English translation).—See *C.A.* 44, 3448*f*. R. J. C.

4-Hydroxy-3-methoxypiperidine. A. Ya. Berlin. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 19, 1177-8 (1949).—Dry distn. of meconic acid with Cu filings gave 50% *2-hydroxy-4-pyrone*, m. 116–18° (from EtOH). This (13 g.) in 100 ml. Et_2O with CH_2N_2 (from 24 ml. $MeN(NO)CO_2Et$) in Et_2O gave *2-methoxy-4-pyrone*, m. 94° (from C_6H_6, after pptn. from $CHCl_3$ by ligroin). This (9 g.), heated 2 hrs. on a steam bath with 75 ml. 25% NH_4OH and 75 ml. H_2O and evapd., gave 8 g. *4-hydroxy-3-methoxypyridine*, needles, m. 174–5° (anhyd.), 114–15° (trihydrate) (from H_2O). This (22 g.) in 250 ml. EtOH treated rapidly with 70 g. Na, then dild. with H_2O, neutralized with HCl, evapd., extd. with dry EtOH, and the ext. treated with 10 g. KOH in abs. EtOH, filtered, and distd. gave 4.90 g. *4-hydroxy-3-methoxypiperidine*, b_{11} 116–17°, b_{17-18} 114–15°, d_4^{25} 1.0770; some 7.35 g. of the corresponding pyridine was recovered. G. M. K.



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1,1'-Dimethyl-2,2'-dihydroxydiethylamine. A. Ya. Berlin and T. P. Sycheva (All Union Chem.-Pharm. Research Inst., Moscow). *Zhur. Obshchei Khim.* (J. Gen. Chem.) **20**, 577-80(1950).—Attempted reduction of $[MeCH(CN)]_2NH$ by H over Raney Ni in the presence of NH_3 failed even at 60° at 15 atm. H; a trace of diamino deriv. formed possibly from EtOH-Na reduction, but the yield was extremely poor. Reductive amination of $AcCH_2OH$ in the presence of NH_3 over Adams Pt oxide also failed; however, 30 g. acetol, 85 ml. 13% NH_4OH, and 5 g. Raney Ni shaken 5 hrs. at 80 (40)° with 15 atm. H gave a small amt. of *bis-acetol di-Me acetal*, $MeO(MeO)CHCH_2CH_2C$-(MeO)$MeCHOCH_3$, m. 120.5 7.0°, and 8 g. fairly pure $MeCH(NH_2)CH_2OH$, b$_{10}$ 70-6° (*phenylthiourea deriv.*, m. 141°). This (1.1 g.) treated with 2 g. BzCl in 5% NaOH gave the *monobenzoate*, m. 104.5 6.0° (from dil. EtOH). Hydrogenation of 7.4 g. $MeCH(NH_2)CH_2OH$ and 0.2 g. acetol over Pt black in MeOH at atm. pressure gave in 4 hrs. 7.8 g. $[MeCH(CH_2OH)]_2NH$, b$_{10}$ 141°, d_4^{25} 1.0140, n_D^{25} 1.4702; *picrate*, yellow plates, m. 105-6° (from EtOH).

G. M. Kosolapoff

CA

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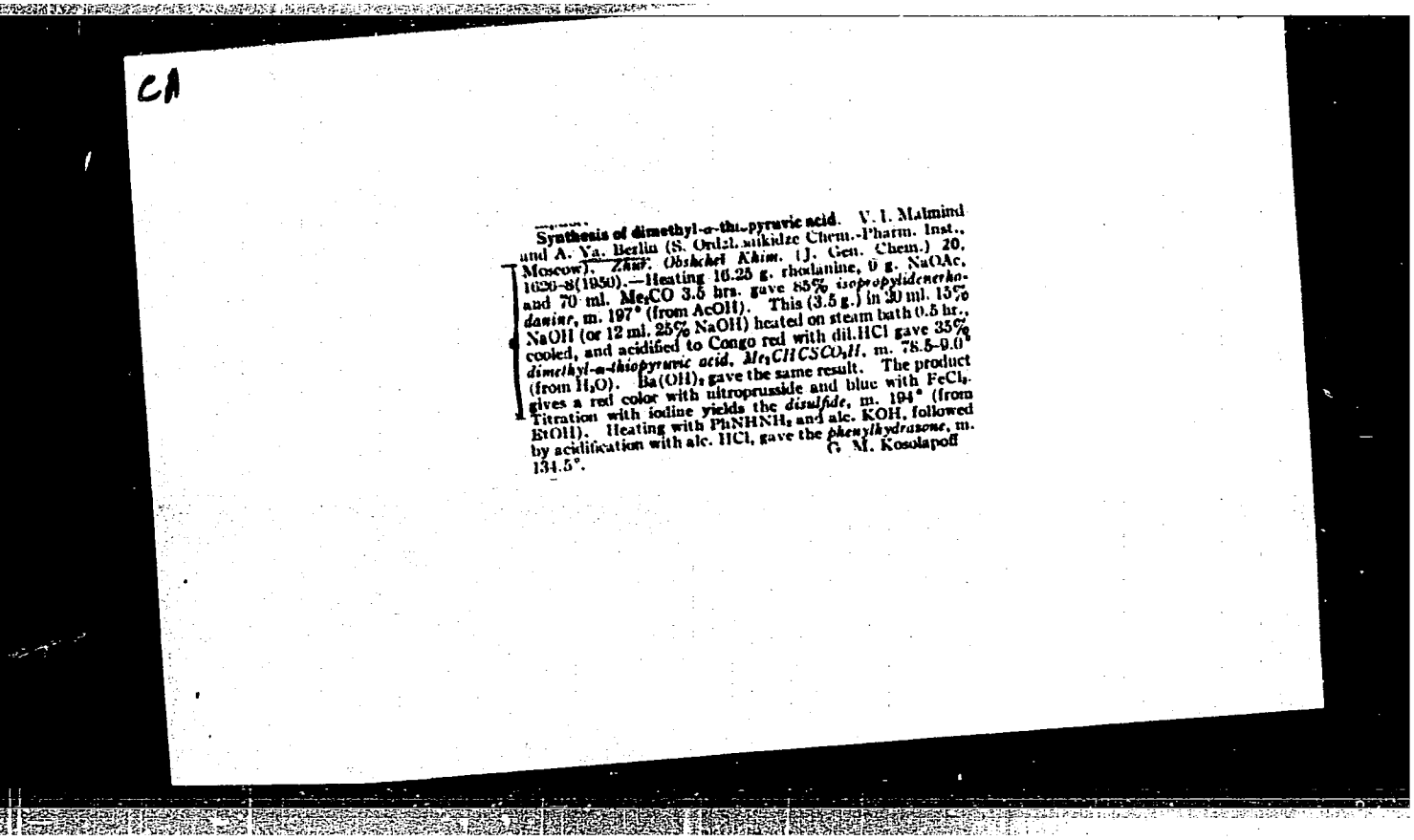
C-Alkyl-substituted morpholines. A. Ya. Berlin and T. P. Sycheva (S. Ordzhonikidze All-Union Chem. Pharm. Research Inst., Moscow). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 640-7(1950).—Addn. of 15 g. propylene oxide in 15 ml. EtOH at 0-5° to 80 g. freshly distd. $H_2NCH_2CH_2OH$ in 100 ml. EtOH gave 66% *HN(CH₂CH₂OH)(CH₂CHMeOH)*, b_6 135°, n_D^{20} 1.4695, d_4^{20} 1.0422; *picrate*, m. 120.5 1.0° (from EtOH-C_6H_6). The product (14.4 g.) carefully added to 12 ml. concd. H_2SO_4 with cooling, followed by 8 hrs. at 170-80° and the usual isolation, gave *2-methylmorpholine-HCl*, which with powd. KOH, gave 80% *free base*, b. 134-6°, n_D^{20} 1.4490, d_4^{20} 0.9581; *phenylthiourea deriv.*, m. 130.5-7.0° (from H_2O). Similarly, 5 g. $H_2NCHMeCH_2OH$ and 1.5 g. ethylene oxide in EtOH gave 95% *HN(CH₂CH₂OH)CHMeCH₂OH*, b_6 131-2°, n_D^{20} 1.4707, d_4^{20} 1.0007; *picrate*, m. 101.5-2.0° (from abs. EtOH-EtOAc). The product treated with H_2SO_4 as above gave 93% *3-methylmorpholine*, b. 123-4°, n_D^{20} 1.4517, d_4^{20} 0.9591; *phenylthiourea deriv.*, m. 121.5-2.5° (from dil. EtOH). Repetition of the above prepn. with propylene oxide (in MeOH) gave 70% *HN(CHMeCH₂OH)(CH₂CHMeOH)*, b_6 140°, n_D^{20} 1.4609, d_4^{20} 1.0129, which similarly yielded 88% *2,5-dimethylmorpholine*, b. 145°, n_D^{20} 1.4459, d_4^{20} 0.9362; *phenylthiourea deriv.*, m. 145-7° (from 50% EtOH). Dehydration by H_2SO_4, as above, of $HN(CHMeCH_2OH)_2$ gave 94% *3,5-dimethylmorpholine*, b. 142.5°, n_D^{20} 1.4460, d_4^{20} 0.9308; *phenylthiourea deriv.*, m. 122-3° (from MeOH). $H_2NCH_2CH_2OH$ (10 g.) with 4 g. *α*-butylene oxide in abs. EtOH gave 61% *HN(CHEtCH₂OH)CH₂CH₂OH*, b_6 137°, n_D^{20} 1.4651, d_4^{20} 1.0115, which on dehydration as above gave 51% *2-ethylmorpholine*, b. 141°, n_D^{20} 1.4490, d_4^{20} 0.9527; *phenylthiourea deriv.*, m. 128-7° (from EtOH). Similarly, $H_2NCHMeCH_2OH$ gave *HN(CHMeCH₂OH)CH₂CHMeOH*, b_6 134-6°, m. 70.6 9.0° (from abs. EtOH); *picrate*, m. 115-16.5° (from Et_2O-EtOH); dehydration, as above, gave 94.6% *2-ethyl-5-methylmorpholine*, b. 162 4°, n_D^{20} 1.4480, d_4^{20} 0.9231; *phenylthiourea deriv.*, m. 117 10° (from dil. EtOH). The use of satd. NH_3 in abs. EtOH in the above reaction gave an unstated yield of *HN(CH₂CH₂OH)₂*, b_6 148 6°, m. 79.5 80.5° (from Et_2O); *picrate*, m. 145 7° (from EtOH); dehydration gave 70% *2,6-diethylmorpholine*, b. 178-9°, n_D^{20} 1.4550, d_4^{20} 0.9175; *phenylthiourea deriv.*, m. 106-7° (from dil. EtOH). The reactions with olefin oxides proceed as well in abs. as in moist alcs., i.e. 95% EtOH.

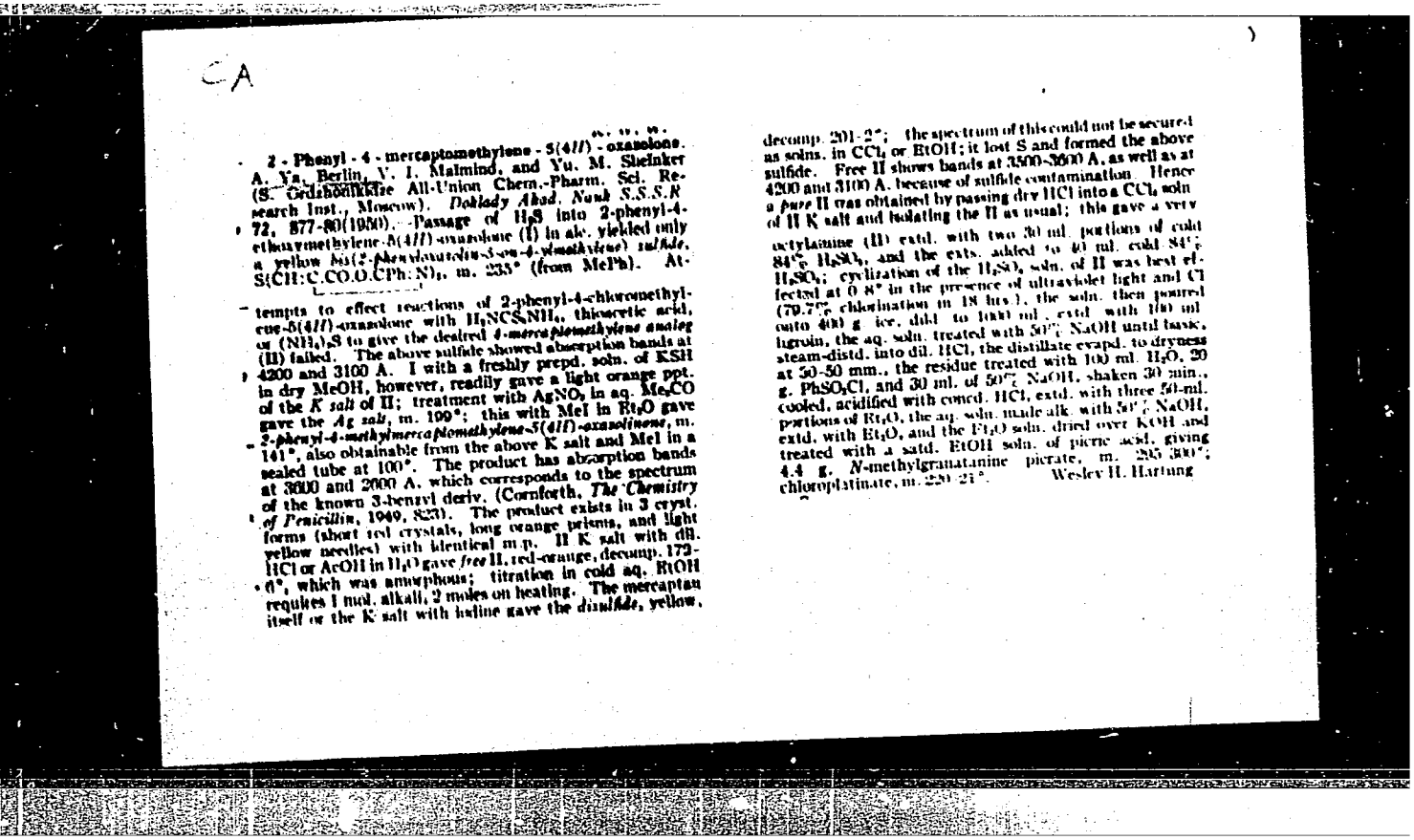
G. M. Kosolapoff



CA

Synthesis of dimethyl-α-thiopyruvic acid. V. I. Malmind and A. Ya. Berlin (S. Ordzhonikidze Chem.-Pharm. Inst., Moscow). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 1630-8(1950).—Heating 16.25 g. rhodanine, 9 g. NaOAc, and 70 ml. Me_2CO 3.5 hrs. gave 85% *isopropylidenerhodanine*, m. 197° (from AcOH). This (3.5 g.) in 20 ml. 15% NaOH (or 12 ml. 25% NaOH) heated on steam bath 0.5 hr., cooled, and acidified to Congo red with dil. HCl gave 35% *dimethyl-α-thiopyruvic acid*, $Me_2CHCSCO_2H$, m. 78.5-9.0° (from H_2O). $Ba(OH)_2$ gave the same result. The product gives a red color with nitroprusside and blue with $FeCl_3$. Titration with iodine yields the *disulfide*, m. 194° (from EtOH). Heating with $PhNHNH_2$ and alc. KOH, followed by acidification with alc. HCl, gave the *phenylhydrazone*, m. 134.5°.

G. M. Kosolapoff



CA

2-Phenyl-4-mercaptomethylene-5(4*H*)-oxazolone. A. Ya. Berlin, V. I. Malmind, and Yu. M. Sheinker (S. Ordzhonikidze All-Union Chem.-Pharm. Sci. Research Inst., Moscow). *Doklady Akad. Nauk S.S.S.R.* 72, 877-80(1950). Passage of H_2S into 2-phenyl-4-ethoxymethylene-5(4*H*)-oxazolone (I) in alc. yielded only a yellow *bis*(2-phenyloxazolin-5-on-4-ylmethylene) *sulfide*, $S(CH{:}C.CO.O.CPh{:}N)_2$, m. 235° (from MePh). Attempts to effect reactions of 2-phenyl-4-chloromethylene-5(4*H*)-oxazolone with $H_2NCS.NH_4$, thioacetic acid, or $(NH_4)_2S$ to give the desired *4-mercaptomethylene analog* (II) failed. The above sulfide showed absorption bands at 4200 and 3100 A. I with a freshly prepd. soln. of KSH in dry MeOH, however, readily gave a light orange ppt. of the *K salt* of II; treatment with $AgNO_3$ in aq. Me_2CO gave the *Ag salt*, m. 199°; this with MeI in Et_2O gave *2-phenyl-4-methylmercaptomethylene-5(4H)-oxazolinone*, m. 141°, also obtainable from the above K salt and MeI in a sealed tube at 100°. The product has absorption bands at 3600 and 2000 A. which corresponds to the spectrum of the known 3-benzyl deriv. (Cornforth, *The Chemistry of Penicillin*, 1949, 823). The product exists in 3 cryst. forms (short red crystals, long orange prisms, and light yellow needles) with identical m.p. II K salt with dil. HCl or AcOH in H_2O gave *free* II, red-orange, decomp. 172-6°, which was amorphous; titration in cold aq. EtOH requires 1 mol. alkali, 2 moles on heating. The mercaptan itself or the K salt with iodine gave the *disulfide*, yellow, decomp. 201-2°; the spectrum of this could not be secured as solns. in CCl_4 or EtOH; it lost S and formed the above sulfide. Free II shows bands at 3500-3600 A. as well as at 4200 and 3100 A. because of sulfide contamination. Hence a *pure* II was obtained by passing dry HCl into a CCl_4 soln. of II K salt and isolating the II as usual; this gave a very

acylamine (II) extd. with two 30 ml. portions of cold 84% H_2SO_4, and the exts. added to 40 ml. cold 84% H_2SO_4; cyclization of the H_2SO_4 soln. of II was best effected at 0-8° in the presence of ultraviolet light and Cl (79.7% chlorination in 18 hrs.), the soln. then poured onto 400 g. ice, dild. to 1000 ml., extd. with 100 ml. lugroin, the aq. soln. treated with 50% NaOH until basic, steam-distd. into dil. HCl, the distillate evapd. to dryness at 50-50 mm., the residue treated with 100 ml. H_2O, 20 g. $PhSO_2Cl$, and 30 ml. of 50% NaOH, shaken 30 min., cooled, acidified with concd. HCl, extd. with three 50-ml. portions of Et_2O, the aq. soln. made alk. with 50% NaOH, extd. with Et_2O, and the Et_2O soln. dried over KOH and treated with a satd. EtOH soln. of picric acid, giving 4.4 g. *N*-methylgranatanine picrate, m. 295-300°; chloroplatinate, m. 220-21°. Wesley H. Hartung

USSR/Chemistry - Synthetic Antibiotics Jan 51

"Methylation of Diethylacetal Formylhyppuric Ester," A. Ya. Berlin, V. I. Maymind, E. S. Golombik, All-Union Sci Res Chemicophar Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol XXI, No 1, pp 132-143

Investigation aimed at synthesis of penicillin-like substances; methylated hippuric ester to form benzoylsarcosine; methylated diethylacetal formalhippuric to obtain (dependent on reaction conditions) ethoxymethylene-N-methylhippuric

173T35

USSR/Chemistry - Synthetic Antibiotics (Contd) Jan 51

ester or 2-phenyloxazol-4-carbonic acid ester, with ethoxymethylenehippuric ester as intermediate product. Ethoxymethylene groups of both ethoxymethylenehippuric ester and ethoxymethylene-N-methylhippuric ester are very resistant to action of alkalis, but only former group can add elements of alc.

173T35

BERLIN, A. YA.

Berlin, A. Ya.: Tekhnika laboratornoi raboty v organicheskoi khimii (The Technique of Laboratory Work in Organic Chemistry). Moscow: State Sci. and Tech. Pub. House Chem. Lit. 1952. 287 pp.

A. Ya. BERLIN

USSR/Chemistry - Pharmaceuticals Sep 52

"Meso-anthranyl-propionic Acids," A. Ya. Berlin, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze

"Zhur Obshch Khim" Vol 22, No 9, pp 1656-1659

It was shown that the condensation of anthracene with acrolein proceeds according to the diene type synthesis and does not require the presence of sulfurous acid as a catalyst. Beta-(anthranyl-9)-acrylic acid and beta-(anthranyl-9)-propionic acid were prepd and characterized, as well as some of their derivs.

232T30

(CA 47 no. 17: 8712 '53)

USSR/Chemistry - Synthetic Drugs Nov 52

"Synthesis of Certain Zingiberone Analogues: V. Derivatives of Resorcinol," A. Ya. Berlin and T. P. Sycheva, All-Union Sci-Res Chem-Pharm Inst imeni S. Ordzhonikidze.

"Zhur Obshch Khim" Vol 22, No 11, pp 1998-2003

The authors were faced with the question of whether the relative positions of the hydroxyl and methoxy groups in the benzene nucleus of compds similar to zingiberone [a] constituent of oil of ginger] had any effect on the physiological action of those

238T31

compds. To determine this, they synthesized a series of compds similar to zingiberone which were derivs of resorcinol and had the hydroxyl and methoxy groups placed in different positions in the nucleus. It was ascertained that these substances had practically no burning taste.

(CA 47 no.17: 8681 '53)

238T31

BERLIN, Ya. A.

USSR/Chemistry - Synthetic Drugs Nov 52

"Synthesis of 1-Methoxyphenanthridine," A. Ya. Berlin and T. P. Sycheva, All-Union Sci-Res Chem-Pharm Inst imeni S. Ordzhonikidze

"Zhur Obshch Khim" Vol 22, No 11, pp 2003-2006

1-methoxyphenanthridine, 1-hydroxyphenanthridine, and a whole series of new derivs of biphenyl were synthesized and described.

238T32

BERLIN, A. Ya.

3

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

(2)

Synthesis of some analogs of zingerone. V. Derivatives of resorcinol. A. Ya. Berlin and T. P. Sycheva. *J. Gen. Chem. U.S.S.R.* 22, 2049-53(1952)(Engl. translation).—See *C.A.* 47, 8881d.

H. L. H.

BERLI , A. Ya.

4

@Chem

Synthesis of 4-methoxyphenanthridine. A. Ya. Berlin and T. P. Sycheva. *J. Gen. Chem. U.S.S.R.* 22, 2055–7 (1952) (Engl. translation).—See *C.A.* 47, 9330b.

H. L. H.

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

June 1952 BERLIN, A. Ya.

Chemistry

Homocyclic

CATALYSTS

Methyl vanillyl sulphide. A. Ya. Berlin. *J. appl. Chem. USSR*, 1952, 25, 566—567).—Vanillyl alcohol is converted into the Bz deriv. (I) by benzoylation in alkaline $COMe_2$-H_2O mixture, in which the product remains in solution and the formation of a large quantity of by-products, as in the usual Schotten–Baumann reaction, is avoided. I is converted into the chloride with $SOCl_2$, and thence to *methyl benzoylvanillyl sulphide* (II), m.p. 79—80°, with MeSNa in C_6H_6-EtOH. Elimination of the Bz group from II by alkaline hydrolysis yields methyl vanillyl sulphide, b.p. 144°/7·5 mm., d^{18} 1·1803.

R. C. MURRAY.

8-31-54 dgp

BERLIN, A. Ya.

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

2
(1) Chem.

Ethyl benzyl ester of methylmalonic acid. A. Ya. Berlin. *J. Appl. Chem. U.S.S.R.* 25, 643–4(1952)(Engl. translation).—See *C.A.* 47, 3241*f*. H. L. H.

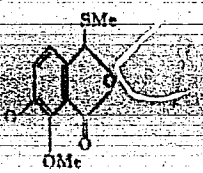
MF

BERLIN, A. YA.

O Chem

Methyl vanillyl sulfide. A. Ya. Berlin. *J. Appl. Chem. U.S.S.R.* 25, 645-0(1952)(Engl. translation).—See *C.A.* 47, 3207d. H.L.H.

Methyl thioester of opianic acid. A. Ya. Berlin (S. Ordzhonikidze All-Union Chem.-Pharm. Inst., Moscow). *Zhurnal Obshchei Khim., Akad. Nauk S.S.S.R.* 1, 640–3 (1953).—Treatment of *opianic acid* with $SOCl_2$ yields the *pseudochloride* of the acid, which, purified by distn., b_{11} 108°; traces of moisture convert the chloride to *opianic anhydride*, m. 230–2° [cf. Liebermann, *Ber.* 19, 2280 (1886); Kanevskaya and Shemyakin, *C.A.* 30, 8425⁴]. The chloride (10 g.) in 200 ml. C_6H_6 was treated with 22 ml. MeSNa soln. contg. 0.149 g. MeSNa per ml.; after 24 hrs., the mixt. was washed with H_2O and the org. layer yielded *Me 3-meconyl sulfide* (I), m. 114–15° (from C_6H_6), which was stable to EtOH, aq. EtOH, or EtOH-HCl. This (1 g.) in 10 ml. AcOH was treated with 3.3 ml. 27% H_2O_2, heated to boiling and allowed to stand overnight, then dild. to 60 ml. with H_2O, yielding a ppt. of 0.83 g. *Me 3-meconyl sulfone* (II), m. 133–4° (from C_6H_6). The structure of II as a deriv. of meconin is confirmed by its formation on treatment of I with H_2O_2. The aldehydo form of I appears to be completely unstable. The ultraviolet spectrum of I in $CHCl_3$ clearly shows its structure as that of a pseudoester of opianic acid [cf. Kirpal, *C.A.* 21, 1642). In EtOH, the spectrum of a fresh soln. of I gradually changes and after several hrs. approaches that of Me opianate. The *O*-contg. ester shows such ionization only in the presence of acids (cf. K. and S., *loc. cit.*).

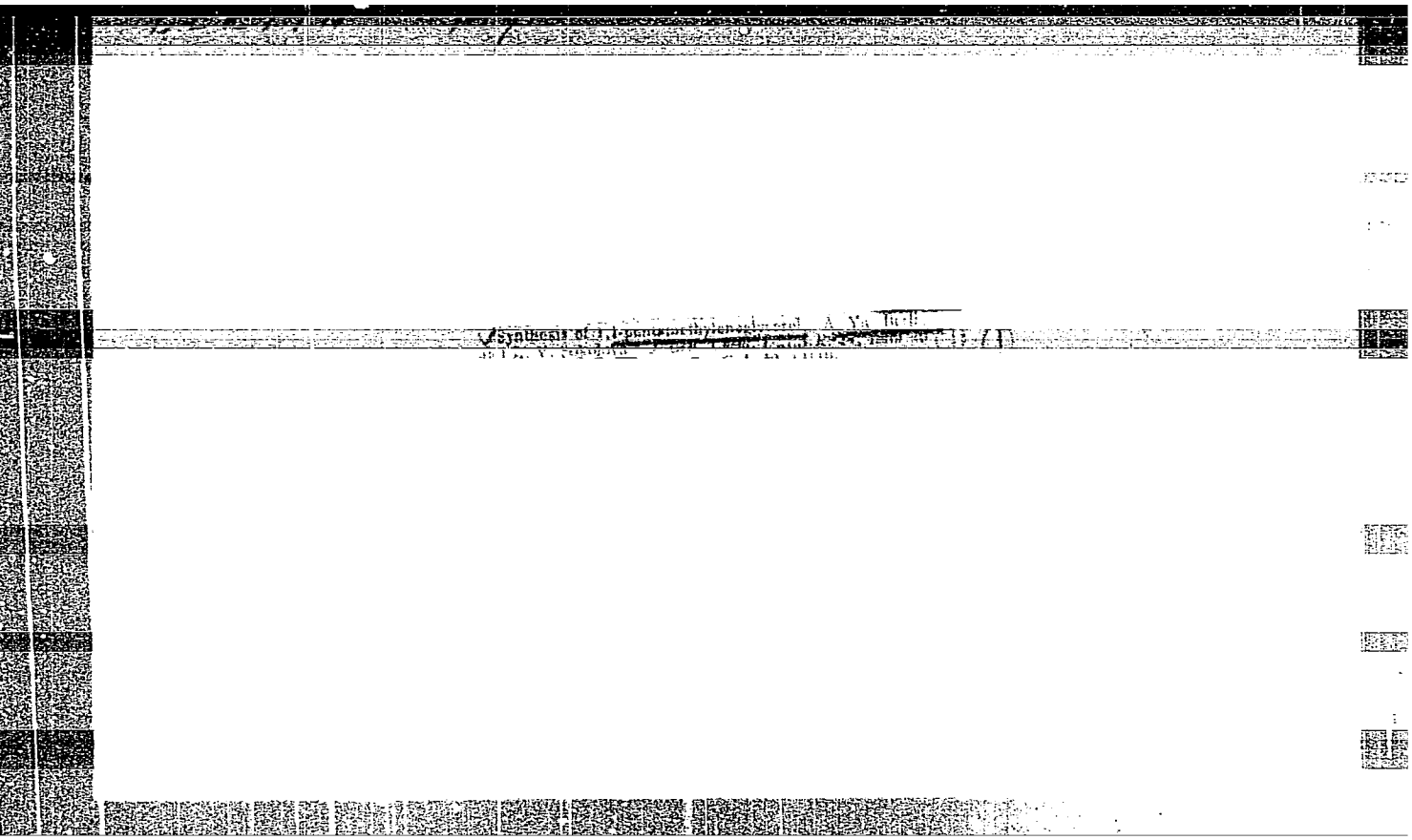


(I)

G. M. Kosolapoff

BERLIN, A. Ya.
VOSKRESENSKIY, P.

"Techniques of laboratory work in organic chemistry." A.IA.Berlin.
Reviewed by P.Voskresenskii. Khim.prom. no.2:127 Mr '54. (MLRA 7:6)
(Chemistry organic--Laberatory manuals)



Berlin, A. Ya.

USSR/Chemistry -- Synthesis methods

Card 1/1 Pub. 151 - 34/37

Authors : Berlin, A. Ya., and Sokolova, L. V.

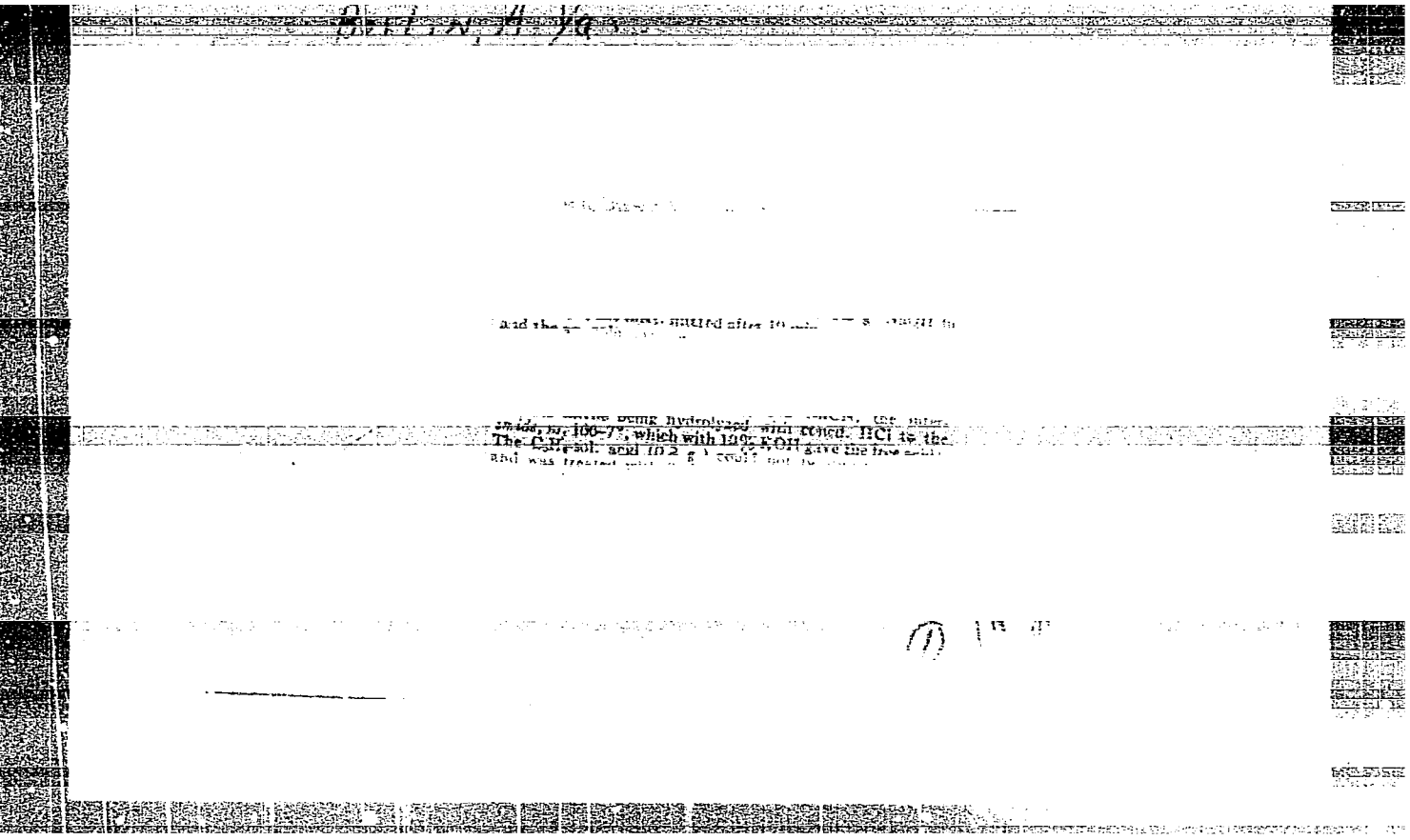
Title : Synthesis of 1,1-pentamethyleneglycerin

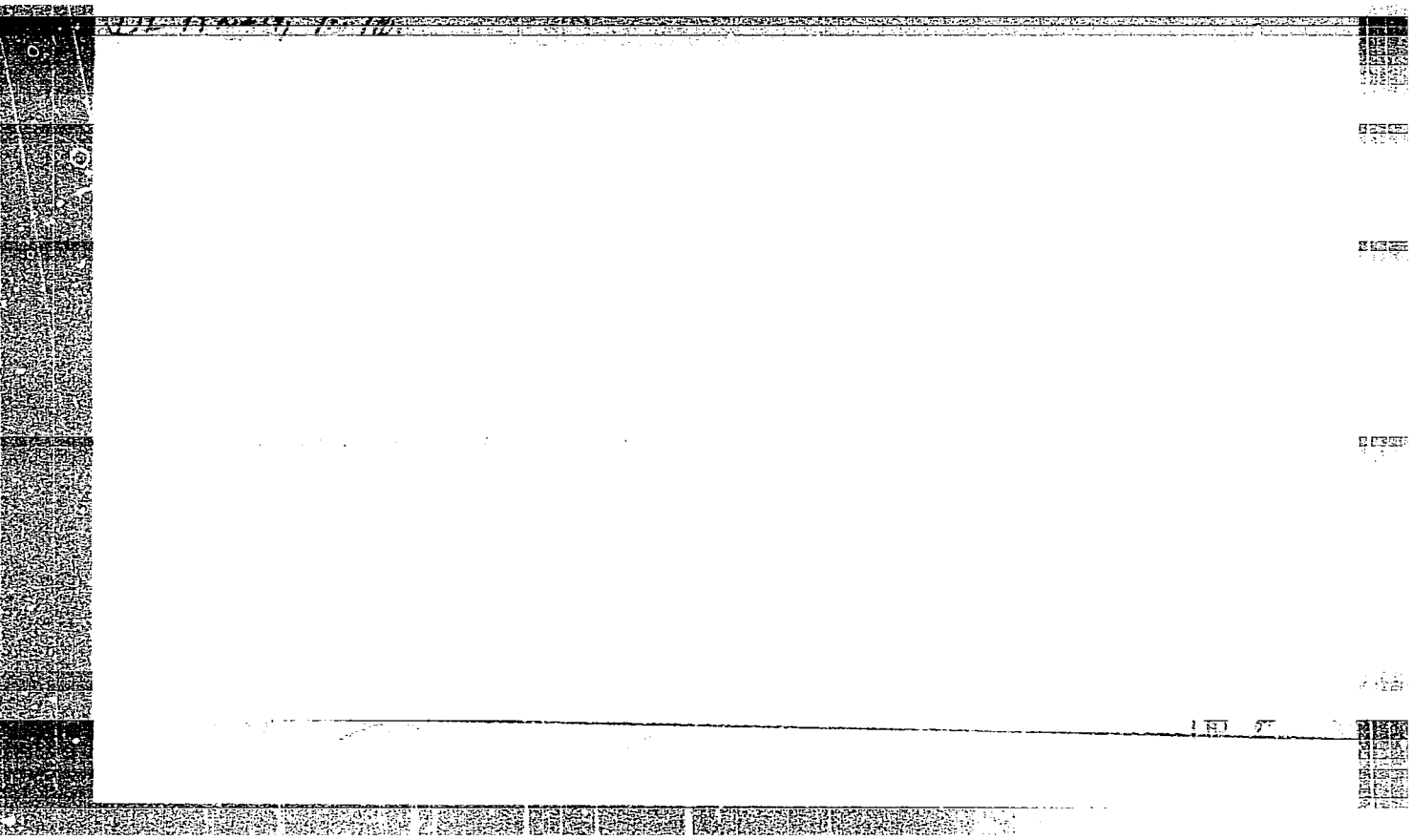
Periodical : Zhur. ob. khim. 24/10, 1874-1884, Oct 1954

Abstract : Two methods employed in the synthesis of 1,1-pentamethyleneglycerin from cyclohexanone are described. Quoting the conversion of ethyl ether of beta,beta-pentamethylene glycidic acid into 3,3-pentamethyleneglycide, as an example, it is shown that glycide ethers can be reduced with lithium alumohydride into homologous alcohols with perfect preservation of the alpha-oxide ring. Ten references: 8-USSR; 1-USA and 1-French (1891-1952).

Institution : The S. Ordzhonikidze All-Union Scientific Research Chemical-Pharmacological Institute

Submitted : April 23, 1954





APPROVED FOR RELEASE: 06/08/2000 CIA-RDP86-00513R000205010001-2"

BERLIN, A.Ya.; SOKOLOVA, L.V.

Formation of ω-acetoxyhexahydroacetophenone and cyclohexylidene-acetaldehyde from 1,1-pentamethyleneglycerin-2,3-diacetate. Zhur. ob.khim.25 no.11:2099-2102 O '55. (MLRA 9:4)

1.Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S.Ordzhonikidze.
(Acetoacetates) (Acetophenone) (Cyclohexaneacetaldehyde)

Berlin, A. Ya.

Substituted glycerin derivatives. [illegible] (na) [illegible] [illegible] 62 586 [illegible] [illegible] acid esters are [illegible] 1,2,3 [illegible] [illegible] 1, 2. The [illegible] are then hydrolyzed in an acid medium. M. Hosch

BERLIN, A.YA.

USSR/General Problems of Pathology - Experimental Therapy. U-3

Abs Jour : Ref Zhur - Biol., No 16, 1958, 75497

Author : Vodolazskaya, N.A., Novikova, M.A., Shkodinskaya, Ye.N., Vasina, O.S., Berlin, A.Ya., Larionov, L.F.

Inst :

Title : On the Antineoplasmic Activity of Some Sarcolysine Derivatives (dl-a-ga-(2-chloroethyl)-aminophenylalanine.

Orig Pub : Byul. eksperim. biol. i med., 1957, 44, No 11, 76-81

Abstract : Toxic and antineoplasmic action (on sarcoma of 85 rats) of 4 sarcolysine derivatives was studied: Ethyl- (I) and isopropyl (II) ethers of dl-sarcolysine, dl-N-formylsarcolysine (III) and dl-N-acetylsarcolysine (IV). It was demonstrated that I and II are very similar to sarcolysine in toxicity and antineoplasmic activity. III and IV are less toxic and their antineoplasmic action is weaker. In order to obtain an effect close to that of sarcolysine,

Card 1/2

it is necessary to take a dose of III 25 times larger than that of sarcolysine (it often produces partial death of animals), and of IV only 1½ to 2 times as large. -- O.V. Zdrave.

Card 2/2

79-28-4-47/60

AUTHORS: Berlin, A. Ya., Vasil'yeva, M. N.

TITLE: Synthesis of the Diethylene-Imide of 4-Methyl Uracil-5--Methylene-Phosphinic Acid (Sintez dietilenimida 4-metiluratsil-5-metilen-fosfinovoy kisloty)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 4, pp. 1063-1065 (USSR)

ABSTRACT: Looking for new chemical means against malignant neoplasms many scientists observed compounds with alkylating effects and containing β,β'-dichlorodiethyl-amino and ethylimino groups. The recently synthesized hydrochloride of p-(β,β'--dichlorodiethyl amino)-phenylalanine (sarcolysin) (Ref 1) is one of the most interesting representatives of this type since its application in medicine made possible for the first time effective treatment of some kinds of genuine tumors in man (Ref 2). The molecule of sarcolysin contains a reactive alkylating dichlorodiethyl amino group combined with the rest of phenylalanine which plays an important part in the albumin metabolism. Looking for compounds of analogous structure and possibly analogous effects the authors synthesized diethylene-imide of the 4-methyl

Card 1/4

Synthesis of the Diethylene-Imide of 4-Methyl Uracil-5-Methylene-Phosphinic Acid

also the hydroxyl groups of the lactim form of the uracil ring (Ref 3) may be exchanged with chlorine. It was found that the reaction between pentoxyl and thionyl chloride is carried out best in chloroform in the cold and under the presence of 1 mol pyridine. A pyridine excess leads to strong resinification. According to the reaction by Arbuzov the diethyl ester of 4-methyl uracil-5-methylene phosphinic acid (III) was produced by the action of triethyl phosphite on compound II. If heated in hydrochloric acid this compound yielded a considerable amount of the corresponding acid (IV). The conversion of this phosphinic acid into its diacid chloride equally made necessary to carry out carefully the reaction since also in this case the already mentioned possibility of unwanted exchange of the hydroxyl groups of the lactim form of the uracil ring with chlorine is given. Even in the case of not rigorous conditions reaction does not take place clearly: a compound of various materials forms from which the acid chloride (IV) could not be separated in its pure form. However, its formation in this reaction is proved, for in the action of anhydrous alcohol on the mentioned compound diethyl ester (III) forms in a

Synthesis of the Diethylene-Imide of 4-Methyl Uracil-5-Methylene-Phosphinic Acid

yield of 21 %. In analogous way diethylene-imide of the 4-methyl uracil-5-methylene phosphinic acid (VI) forms a crystallized compound during the action of ethyleneimine on the reaction product of the phosphinic acid (IV) with thionyl chloride (Ref 6), which changed when heated in a capillary without showing a certain melting point. M. I. Kabachnik and T. Ya. Medved' kindly devoted themselves to the described works.

The method of synthesis is described in detail in an experimental chapter. There are 6 references, 2 of which are Soviet.

SUBMITTED: March 27, 1957

Card 4/4

VODOLAZSKAYA, N.A., NOVIKOVA, M.A., SHKODINSKAYA, Ye.N., VASINA, O.S. BERLIN, A.Ya., LARIONOV, L.F.

Anti-tumor effect of certain sarcolysin derivatives; dl-P-di-(chloroethyl) aminophenyl-lalanine [with summary in English] Biul.eksp.biol. i med. 44 no.11:76-81 Jl-Ag '58 (MIRA 11:11)

1. Iz laboratorii eksperimental'noy khimioterapii (zav. - chlen-korrespondent AMN SSSR L.F. Larionov) i laboratorii khimicheskogo sinteza (zav. - prof. A.Ya. Berlin) Instituta eksperimental'noy patologii i terapii raka (dir. - chlen-korrespondent AMN SSSR N.N. Blokhin) AMN SSSR, Moskva. Predstavlena deystvitel'nym chlenom AMN SSSR V.V. Zakusovym.

(NITROGEN MUSTARDS, effect,
dl-p-di- (Chloroethyl) aminophonylalanine,
on exper. spindle cell sarcoma (Rus))
(SARCOMA, experimental,
dl-p-di- (chloroethyl) aminophenylalanine (Rus))

BERLIN, A.Ya. (Moskva, G-34, Kropotkinskiy per., d.25, kv.29)

Evaluation of the antitumor activity of chemical preparations. Vop. onk. 5 no.9:346-350 '59. (MIRA 12:12)

1. Institut eksperimental'noy i klinicheskoy onkologii AMN SSSR (dir. - chlen-korrespondent AMN SSSR prof. N.N. Blokhin).
(ANTINEOPLASTIC AGENTS ther.)

ASTRAKHAN, V.I., doktor med.nauk; BERLIN, A.Ya., prof.; IAZAREV, N.I., kand.biologicheskikh nauk; PEREVODCHIKOVA, N.I., kand.med.nauk

Second Coordinating Conference on Chemotherapy in Cancer. Vest. AMN SSSR 14 no.5:77-82 '59. (MIRA 14:5)
(CANCER—CONGRESSES)

AUTHORS: Makarova, A. N., Berlin, A. Ya. SOV/79-29-2-64/71

TITLE: Reaction of Ethylene Imino Benzoquinones-1,4 With Amines (Vzaimodeystviye etileniminobenzokhinonov-1,4 s aminami). I. Reaction of Ethylene Imino Benzoquinone-1,4 With Secondary Amines (I. Reaktsiya mezhdu etilenimi nobenzokhinonami-1,4 i vtorichnymi aminami)

PERIODICAL: Zhurnal obshchoy khimii, 1959, Vol 29, Nr 2, pp 666-672 (USSR)

ABSTRACT: The task of the work under review was the reaction of 2,5-diethylene imino benzoquinone-1,4, as well as of 2,5-dichloro and 2,5-diethoxy-3,6-diethylene imino benzoquinone-1,4 with secondary amines. The reaction of ethylene imino quinones with secondary amines may take place in two directions (Scheme). In most cases it proceeds smoothly and in good yields on briefly heating the diethylene imino quinones with an excess of amine in the methanol medium or without solvent. Only in the reaction of 2,5-diethylene imino quinone and 2,5-dichloro--3,6-diethylene imino quinone with diethyl amine, ammonium chloride was used as catalyst. Experimental conditions and the compounds synthesized in this connection are specified in table 1, and their physical properties in table 2.

Card 1/2

Reaction of Ethylene Imino Benzoquinones-1,4 With Amines. I. Reaction of Ethylene Imino Benzoquinone -1,4 With Secondary Amines

SOV/79-29-2-64/71

6 new compounds were synthesized. It was found that on the reaction of ethylene imino quinones with amines, cleavage products of the ethylene imino cycle of the bis-(alkylamino ethylamino)-quinone-type are formed and also products of the substitution of ethylene imino radicals by those taken in the reaction of secondary amines were found to occur. It was shown that the facility of the cleavage of the ethylene imino cycle in ethylene imino quinones depends on the character of the substituents in the quinone nucleus. There are 2 tables and 20 references, 6 of which are Soviet.

ASSOCIATION: Institut eksperimental'noy patologii i terapii raka Akademii meditsinskikh nauk (Institute of Experimental Pathology and Cancer Therapy of the Academy of Medical Sciences)

SUBMITTED: December 23, 1957

Card 2/2

5 (3)
AUTHOR: Berlin, A. Ya.

SOV/79-29-7-64/83

TITLE: On Some Reactions of β,β'-Dioxydiethylamino-n-benzoquinone (O nekotorykh reaktsiyakh β,β'-dioksidietilamino-n-benzokhinona)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 7, pp 2390 - 2394 (USSR)

ABSTRACT: Condensation of n-benzoquinone with amines yielded the corresponding derivative of 2,5-diaminoquinone in nearly all cases (Ref 1). However, the authors obtained only β,β'-dioxydiethylamino-n-benzoquinone (I) in 90% yield when they treated n-benzoquinone with diethanolamine in alcohol ether solution. The second diethanolamino group did not enter the benzoquinone nucleus. The substance (I) exhibited some special properties: mineral acids, for instance, changed the color of its solution to dark purple. Some interesting observations were made on attempting to reduce (I) to (II): treatment of compound (I) with zinc dust in weak acetic acid yielded a product melting at 106-107° and having the empirical formula $C_{16}H_{13}O_3N$. Of the three structural formulas possible (III) is the most likely one.

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By reduction of the above dark purple solution of (I) with 2-3% hydrochloric acid and zinc dust (or SO_2) two bases were obtained which could be separated in hydrochloric acid (1:1) due to their different solubility. One of them, with the empirical formula $C_{10}H_{16}O_3NCl$ undoubtedly had the structure (VII) and was the product of a cyclization, resulting from the elimination of water from the monochlorine derivative of the dioxydiethylaminohydroquinone. (VII) was formed by the addition of HCl to (I) to close then the morpholine ring (Reaction Scheme 1). Thus, zinc dust as a reducing agent was superfluous, as was proved by a further experiment using hydrochloric acid alone for the conversion of (I) to (VII). The second base contained no chlorine and had the empirical formula $C_{20}H_{24}O_6N_2$. There are 8 references, 2 of which are Soviet.

On Some Reactions of β,β'-Dioxydiethylamino-n-benzoquinone SOV/79-29-7-64/83

ASSOCIATION: Institut eksperimental'noy patologii i terapii raka Akademii meditsinskikh nauk SSSR, Moskva (Moscow Institute of Experimental Pathology and Therapy of Cancer of the Academy of Medical Sciences, USSR)

SUBMITTED: May 4, 1958

Card 3/3

SOV/20-126-4-31/62

5(3)

AUTHORS: Chernova, N. G., Yaguzhinskiy, L. S., Berlin, A. Ya.

TITLE: The Synthesis of β-(p-di-(2-Chloroethyl)-aminophenyl)-β-alanine (Sintez β-(p-di-(2-khloretil)-aminofenil)-β-alanina)

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 126, Nr 4, pp 802-805 (USSR)

ABSTRACT: As is known, "Sarcolysine" (p-di-(2-chloroethyl)-amino-β-phenyl-2-alanine) possesses a high anti-tumor activity in the experiment as well as in the clinic (Refs 1, 2). It therefore was of interest for the authors to synthesize the chemically related substance, as mentioned in the title (I). It is a derivative of β-amino acid. β-(p-nitrophenyl)-β-N-acetyl-β-alanine (II), produced according to V. M. Rodionov's method, served as initial substance. Since the synthesis was difficult, due to a protection of the β-amino group by the rest of acetyl, and as the output was small (15%) a second way was studied: with a phthaloyl protection of the β-amino group. It proved completely satisfactory. The first way is described. Investigating the second way, β-(p-nitrophenyl)-β-alanine (VII) (Ref 3) was used as initial substance. It was esterized by means of an alcoholic HCl solution. A successive treatment with phthalic acid anhydride and acetic acid anhydride (Ref 5) converted the β-(p-nitrophenyl)-β-alanine-ethylester (VIII)

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The Synthesis of β-(p-di-(2-Chloroethyl)-aminophenyl)-β-alanine

immediately into β-(p-nitrophenyl)-β-N-phthaloyl-alanine-ethylester (IX). (IX) was synthesized into β-(p-aminophenyl)-β-N-phthaloyl-β-alanine-ethylester (X) by means of hydration in the presence of skeleton nickel. Analogous to the transformations of (IV) into (I), several successive syntheses of a phthaloyl compound (X) were carried out without isolating the intermediate products: β-(p-di-(2-oxyethyl)-aminophenyl)-β-N-phthaloyl-β-alanine-ethylester (XI) (Ref 6), β-(p-di-(2-chloroethyl)-aminophenyl)-β-N-phthaloyl-β-alanine-ethylester (XII), chlorine hydrate (I) as well as base (I). The latter was produced with a yield of 48%. There are 6 references, 3 of which are Soviet.

ASSOCIATION: Institut eksperimental'noy patologii i terapii raka Akademii meditsinskikh nauk SSSR (Institute of Experimental Pathology and Cancer Therapy of the Academy of Medical Sciences, USSR)

PRESENTED: February 7, 1959, by M. M. Shemyakin, Academician

SUBMITTED: January 13, 1959

5.3610

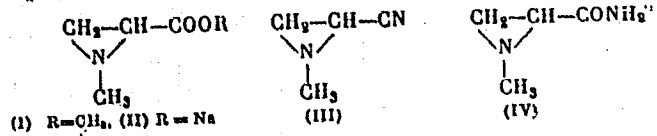
77371
SOV/79-30-1-32/78

AUTHORS: Antonov, V. K., Berlin, A. Ya.

TITLE: Alkaline Saponification of Esters and Nitriles of Ethyleneiminocarboxylic Acids

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 1, pp 151-153 (USSR)

ABSTRACT: The methyl ester of N-methylethyleneiminocarboxylic acid (I) was saponified with alcoholic NaOH, and only one product, the sodium salt of N-methylethyleneiminocarboxylic acid (II) was isolated, in 25% yield, mp 222-223° (dec). The free acid was not obtained by acidifying the above salt, but rather a water soluble polymeric product was obtained.



(I) R=CH₃, (II) R = Na (III) (IV)

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Alkaline Saponification of Esters and Nitriles of Ethyleneiminocarboxylic Acids

77371
SOV/79-30-1-32/78

The amide of N-methylethyleneiminocarboxylic acid (IV) was obtained in 25% yield (mp 100-101°) by saponification of the nitrile of N-methylethyleneiminocarboxylic acid (III) with aqueous-alcoholic alkali. The corresponding sodium salt (II) was obtained in 40-45% yield. At the same time amide (IV) was obtained, in 27.5% yield (mp 98-100°), by saponification of nitrile (III) with a solution of KOH containing 3% H_2O_2. There are 4 references, 1 Soviet, 1 German, 2 U.S. The U.S. references are: M. A. Stolberg, J. O'Neill, T. Wagner-Jauregg, J. Am. Chem. Soc., 75, 5045 (1953); G. Jones, J. Org. Chem, 9, 125 (1944).

SUBMITTED: December 17, 1958

Card 2/2

5.3610

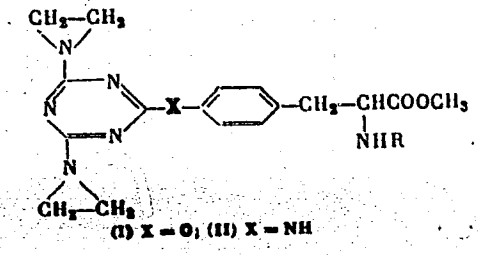
77401
SOV/79-30-1-62/78

AUTHORS: Berlin, A. Ya., Antonov, V. K.

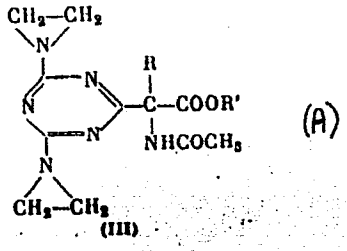
TITLE: Some Diethyleneiminotriazine Derivatives of α-Amino Acids

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 1, pp 282-286 (USSR)

ABSTRACT: Analogs of the toxic carcinostatic drug, triethylene-imino-S-triazine (TET), diethyleneiminotriazine compounds of type (I), (II), and (III), containing radicals of α-amino acid, were prepared and tested as drugs.



(I) X = O; (II) X = NH



(III) (A)

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Some Diethyleneiminotriazine Derivatives of α-Amino Acids

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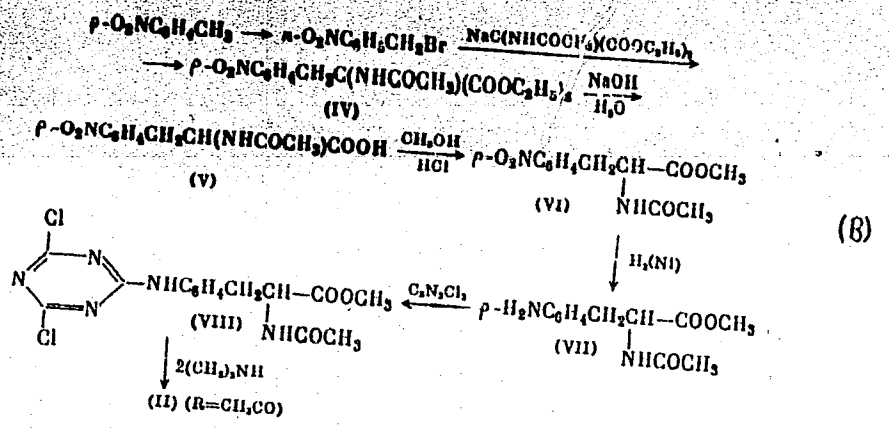
SOV/79-30-1-52/78



(8)

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Some Diethyleneiminotriazine Derivatives of α -Amino Acids

77401
SOV/79-30-1-62/78

ASSOCIATION: Institute of Experimental Pathology and Therapy of Cancer, Academy of Medical Sciences USSR (Institut eksperimental'noy patalogii i terapii raka Akademii meditsinskikh nauk SSSR)

SUBMITTED: December 17, 1958

Card 4/4

5.3900

77410
SOV/79-30-1-71/78

AUTHORS: Berlin, A. Ya., Bronovitskaya, V. P.

TITLE: p-Bis-(2-Chloroethyl)-Aminophenylalanine (Sarcolysin) and Its Derivatives. V. Heterocyclic Amides of Sarcolysin

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 1, pp 324-327 (USSR)

ABSTRACT: Some of the p-bis-(2-chloroethyl)aminophenylalanylpeptides have, like sarcolysin, anticancerous properties, without having its toxicity. In view of this, N-acetylsarcolysin (thiazolyl-2)amide (I), N-acetylsarcolysin (4-methylthiazolyl-2)amide (II), N-acetylsarcolysin (piperidyl)amide (III), N-acetylsarcolysin (morpholyl) amide (IV), and N-formylsarcolysin (thiazolyl-2)amide (V) were synthesized by successive addition of equimolar quantities of 1,3-dicyclohexylcarbodiimide and corresponding heterocyclic amine in chloroform to a chloroform suspension of 0.01 mole of N-acylsarcolysine

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p-Bis-(2-Chloroethyl)-Aminophenylaline (Sarcolysin) and Its Derivatives. V. Heterocyclic Amides of Sarcolysin

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SOV/79-30-1-71/78

(method of Sheehan (Sheehan, J. C., Hess, G., J. Am. Chem. Soc., 77, 1067 (1955)). The reaction mixture was left at room temperature for 5 hr (except in preparation of compound V, when only 30 min was necessary) and filtered to separate the amide solution from the 1,3-dicyclohexylurea. The amide separated on the second day from the filtrate (or crystallized out after distilling the chloroform and adding absolute alcohol with subsequent cooling) and was recrystallized from absolute alcohol. Table A gives the yields and melting points of the compounds along with the preparation scheme for the first four. Since, according to F. Bergel and J. A. Stock (J. Chem. Soc., 1957, 4563; Proc. Roy. Soc., 1957, 60), a free amino-group in the sarcolysin compound is essential for anticancerous properties, the authors synthesized sarcolysin (thiazolyl-2)amide (VI) (by hydrolysis of N-formylsarcolysin (thiasolyl-2)amide (V)).

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p-Bis-(2-Chloroethyl)-Aminophenylaline (Sarcolysin) and Its Derivatives. V. Heterocyclic Amides of Sarcolysin

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Table A. Heterocyclic amides of sarcolysin.

COMPOUND	EMPIRICAL FORMULA	YIELD (%)	MELTING POINT	FOUND (%)				CALCULATED (%)			
				C	H	N	Cl	C	H	N	Cl
(I) N-ACETYLSARCOLYSIN (THIAZOLYL-2) AMIDE (I)	$C_{12}H_{13}O_2N_4Cl_2S$	52.2	165.5–166.5°	50.35	5.04	12.62	16.53	50.35	5.13	13.05	16.55
(II) N-ACETYLSARCOLYSIN-(4-METHYLTHIAZOLYL-2)-AMIDE (II)	$C_{13}H_{15}O_2N_4Cl_2S$	52.2	183–184	51.42	5.52	12.29	16.04	51.46	5.41	12.60	16.00
(III) N-ACETYLSARCOLYSIN (PIPERIDYL) AMIDE (III)	$C_{20}H_{30}O_2N_3Cl_2$	57.4	148–149	57.95	7.03	10.45	16.89	57.97	7.00	10.14	17.15
(IV) N-ACETYLSARCOLYSIN (MORPHOLYL) AMIDE (IV)	$C_{19}H_{27}O_3N_3Cl_2$	65.2	155–156	54.28	6.66	10.17	17.11	54.80	6.49	10.09	17.08
(V) N-FORMYLSARCOLYSIN (THIAZOLYL-2) AMIDE (V)	$C_{17}H_{20}O_2N_4Cl_2S$	80.5	170–171	49.15	4.81	12.93	16.86	49.15	4.82	13.49	17.10

(cont. next card)

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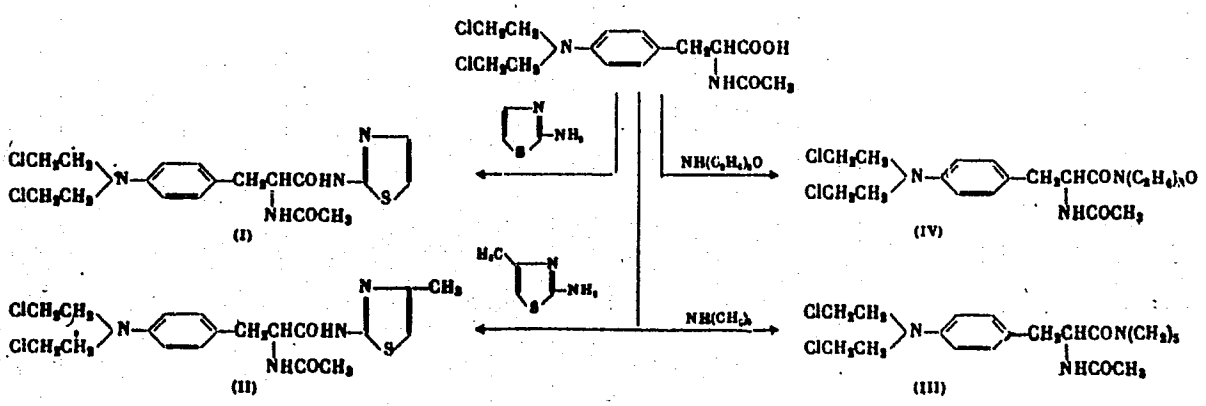
77410, SOV/79-30-1-71/78

TABLE A (cont.)



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p-Bis-(2-Chloroethyl)-Aminophenylaline (Sarcolysin) and Its Derivatives. V. Heterocyclic Amides of Sarcolysin

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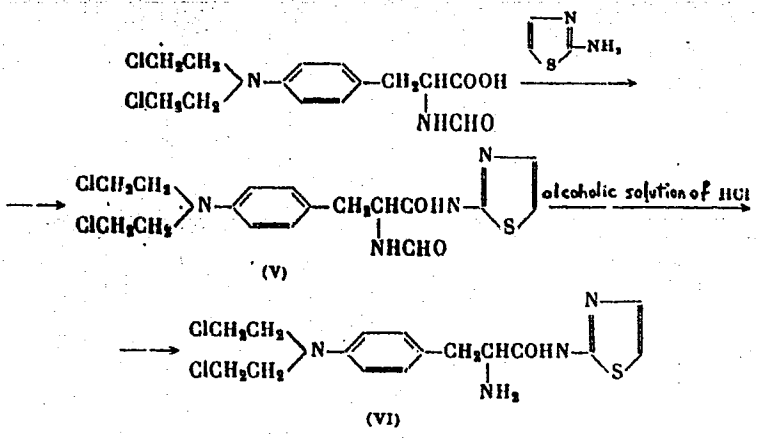
SOV/79-30-1-71/78

Preparation scheme for V and VI is shown below:



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p-Bis-(2-Chloroethyl)-Aminophenylaline (Sarcolysin) and Its Derivatives. V. Heterocyclic Amides of Sarcolysin

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Compound VI was prepared by dissolving 2.2 g of V in 300 ml of an alcoholic solution of 1N HCl and, after letting the solution stand at room temperature for 1 hr, concentrating it under vacuum to a small volume. The precipitate was filtered off and recrystallized from absolute alcohol (Yield 68%; mp 226-227°). The results of biological study of the synthesized preparates will be published elsewhere. There are 1 table; and 6 references, 3 Soviet, 2 U.K., 1 U.S. The U.S. and U.K. references are: J. C. Sheehan, G. Hess, J. Am. Chem. Soc., 77, 1067 (1955); F. Bergel, J. A. Stock, J. Chem. Soc., 1957, 4563; Pr. Roy. Soc., 1957, 60; S. Waley, Chem. and Ind., 1953, 107.

SUBMITTED: November 3, 1958

Card 6/6

S/079/60/030/04/76/080
B001/B003

AUTHORS: Berlin, A. Ya., Makarova, A. N.

TITLE: Reaction of Ethoxychloroquinone With Amines. I. Reactions of Diethoxydichlorobenzoquinone-1,4

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 4, pp. 1380-1385

TEXT: In continuation of Refs. 6-8 regarding the formation of the derivatives of 2,5-diamino-3,6-dichlorobenzoquinone in the article under review certain interesting facts were discovered in the investigation of the reaction of 2,5-diethoxy-3,6-dichloro-benzoquinone and 2,6-diethoxy-3,5-dichlorobenzoquinone with amines. Until now, no derivatives of the 2,6-diaminobenzoquinone or 2,6-diamino-3,5-dichlorobenzoquinone were obtained (Refs. 9-11). The 2,5-diethoxy-3,6-dichlorobenzoquinone-1,4 and 2,6-diethoxy-3,5-dichlorobenzoquinone-1,4 compounds required for the investigation were obtained by heating an alcoholic suspension of chloranil in the presence of triethylamine in a molar ratio of 1:2 between chloranil and triethylamine in a ratio of 1:1 a mixture of all three ethoxychlorobenzoquinones results. Quinone (IV) was obtained by the re-

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action of 2,5 dichloro-3,6-dichloroquinone with ethylene imine which also results from chloranil and ethylene imine (Ref. 3). Quinone (V) (Scheme) also results from 2,6-diethoxy-3,5-dichloroquinone. On the strength of previous experience (Refs. 12,13) the authors utilized the reaction of 2,5-diethylene iminobenzoquinone with various amines in order to obtain the derivatives of the 2,6-diamino-3,5-dichloroquinone. When compound (V) is reacted with benzylamine, cyclohexylamine and morpholine a new interesting kind of regrouping is additionally determined. Instead of the derivatives of 2,6-diamino-3,5-dichlorobenzoquinone derivatives of 2,5-diamino-3,6-dichlorobenzoquinone (VI, VII and VIII) formed, i.e., the same compounds which were obtained from compound (IV) or from (I) and the amines indicated. Thus, 2,5-diethyleneimino-3,6-dichlorobenzoquinone-1,4 and 2,6-diethyleneimino-3,5-dichlorobenzoquinone-1,4 were synthesized in the reaction of 2,5-diethoxy-3,6-dichlorobenzoquinone and of the 2,6-diethoxy-3,5-dichlorobenzoquinone with ethyleneimine. There are 1 table and 15 references, 3 of which are Soviet.

V

SUBMITTED: March 20, 1959

Card 2/2

MAKAROVA, A.N.; GRIBKOVA, M.P.; BERLIN, A.Ya.

Interaction between acyloxydichloro-p-benzoquinones and amines.
Zhur.ob.khim. 30 no.5:1577-1581 ~1960 (MIRA 13:5)

1. Institut eksperimental'noy i klinicheskoy onkologii Akademii meditsinskikh nauk SSSR.
(Benzoquinone) (Amines)

BERLIN, A.Ya.; MAKAROVA, A.N.

Interaction between ethoxychloroquinones and amines. Part 2: Reactions of monoethoxytrichloro-p-benzoquinone. Zhur.ob. khim. 30 no.5:1582-1585 My '60. (MIRA 13:5)

1. Institut eksperimental'noy i klinicheskoy onkologii Akademii meditsinskikh nauk SSSR.
(Benzoquinone) (Amines)

BERLIN, A.Ya.; ZAYTSEVA, V.N.

Cyclization of substituted phenyl hydrazones of ethyl α-keto-γ-diethylaminobutyrate by means of. Zhur.ob.khim. 30 no.7: 2368-2371 Jl '60. (MIRA 13:7)

1. Institut eksperimental'noy i klinicheskoy onkologii Akademii meditsinskikh nauk SSSR.
(Hydrazones) (Butyric acid) (Indole)

BERLIN, A.Ya.; URETSKAYA, G.Ya.; RYBKINA, Ye.I.

New type of disproportionation. Zhur. ob. khim. 30 no.12:4109-4110 D '60. (MIRA 13:12)

1. Institut eksperimental'noy i klinicheskoy onkologii Akademii meditsinskikh nauk SSSR.
(Disproportionation)

VASIL'YEVA, M.M.; SHKODINSKAYA, Ye.N.; BERLIN, A. Ya.

Sarcolysine isomers and their derivatives. Part 2: Synthesis of o-bis (2-chloroethyl)amino-DL-phenylalanine. Zhur. ob. khim. 31 no.3:1027-1033 Mr '61. (MIRA 14:3)

1. Institut eksperimental'noy klinicheskoy onkologii AMN SSSR.
(Alanine)

BERLIN, A. Ya.; BRONOVITSKAYA, V.P.

p -Di(2-chloroethyl)-aminophenylalanine ("sarcolysin") and its derivatives. Part 6: Amides from N-acetylsarcolysine and some amines of the thiazole series. Zhur. ob. khim. 31 no.4:1356-1361 Ap '61. (MIRA 14:4)

1. Institut eksperimental'noy i klinicheskoy onkologii Akademii meditsinskikh nauk SSSR.
(Amines)
(Sarcolysin)

MAKAROVA, A.N.; BERLIN, A.Ya.

Interaction of ethyleneiminochloro-1,4-benzoquinones with α-alanine ethers. Zhur.ob.khim. 31 no.7:2353-2358 Jl '61. (MIRA 14:7)

1. Institut eksperimental'noy i klinicheskoy onkologii Akademii meditsinskikh nauk SSSR.
(Benzoquinone) (Alanine)

SHKODINSKAYA, Ye.N.; KURDYUKOVA, Ye.M.; BERLIN, A.Ya.

p-Di-(2-chloroethyl)-amino-dl-phenylalanine (sarcolysine) and its derivatives. Part 7: Halogen-substituted in the ring sarcolysine derivatives. Zhur. ob. khim. 31 no. 11:3788-3793 N '61. (MIRA 14:11)

1. Institut eksperimental'noy i klinicheskoy onkologii Akademii meditsinskikh nauk SSSR.

(Sarcolysine)

BERLIN, A.Ya., red.; KUZ'MINA, N.S., tekhn. red.

[Ways for synthesizing and testing antineoplastic preparations; transactions] Puti sinteza i izyskaniia protivoopukholevykh preparatov; trudy. Pod red. A.IA.Berlina. Moskva, Medgiz, 1962. 211 p. (MIRA 15:6)

1. Simpozium po khimii protivoopukholevykh veshchestv, Moskva, 1960.

(CYTOTOXIC DRUGS)

L 12341-63 EWT(m)/BDS RM S/081/63/000/005/033/075 53

AUTHOR: Makarova, A. N., Gribkova, M. P. Martynov, V. S. and Berlin, A. Ya.

TITLE: Substitution reactions in a series of derivatives of benzoquinone-1,4

PERIODICAL: Referativnyy zhurnal, Khimiya, no. 5, 1963, 203-204, abstract 5Zh131 (Puti sinteza i izyskaniya protivoopukholevykh preparatov, M, Medgiz, 1962, 165-174)

TEXT: Substitution reactions were investigated of functional groups by the amino-groups in 2,5-diethylenimino-3-R-6-R'- benzoquinones-1,4 (I), 2,6-diethylenimino-3,5-dichlorbenzoquinone-1,4 (II) and 6-monoethylenimino-2,3,5-trichlorbenzoquinone-1,4 (III). In almost all cases anomalous trends were discovered in the reactions. Thus, in treating I with primary amines $R''NH_2$ a substitution of ethylenimino groups by amino groups occurs with formation of corresponding 2,5$(R''NH)_2$-3-R-6-R'-benzoquinones-1,4 (IV). The speed of reaction depends, to a significant degree, on the nature of the replacements and on the basic characteristics of the amines. The following IV were obtained (below are given R, R', R'', time of reaction in min, yield of IV in % and m.p. in °C): H, H, iso-C_3H_7, 40, 90, 240 - 241; H, H, C_6H_{11}, 18, 94, 239 - 240; H, H, C_6CH_2, 10, 80, 250 - 251; H, Cl iso-C_3H_7,

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L 12341-63 S/081/63/000/005/033/075

Substitution reactions in ....

20, 63, 157 - 158; H, Cl, $C_6H_5CH_2$, 3, 80, 207 - 208; H, OC_2H_5, $C_6H_5CH_2$, 440, 34, 204 - 205; Cl, Cl, iso-C_3H_7, 30, 95, 200 - 223; Cl, Cl, C_6H_{11} (IVa), 13, 93, 233 - 234; Cl, Cl, $C_6H_5CH_2$ (IVb), 5, 90, 220 - 223; Cl, Cl, C_6H_5, 120, 55, 285 - 286.

II reacted in the same manner, but significantly slower. Concurrently, the exchange of the atoms of chlorine with amine groups occurred, leading to IVa, b with yields of 50 and 30% respectively. The regrouping mechanism was not studied. Only in the case of III initially or concurrently with the replacement of the ethylenimino group the replacement of the Cl atom by amine groups occurred with formation of 2-ethylenimino-5-(N-morpholinyl)- or 2 ethylenimino-5-cyclohexylamino-3,6-dichlorquinone. Already at 20°C it appeared possible to obtain satisfactory yields of reaction products. The same behavior was confirmed on the example of reactions of I - III with methyl or ethyl ester of α -analine (V). However, fluoranalogs of I - III under the same conditions disclosed considerable mobility of the F atom, sufficient, for preparative purposes. In the treatment of aniline fluoride with 4 moles of ethylenimine (VI), V or ethyl ester of α -phenyl-β -aniline were obtained (here and henceforth are shown the substance, the yield in %, and m.p. in °C): 2,5-diethylenimino-3,6-difluorquinone (VII), 72, 211 - 213; diethyl ester of 2,5-

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Substitution reactions in .....

di-(N-alanino)- 3,6 difluorquinone, 75, 132 - 133 and 178 - 179 (dimorphism); diethyl ester of 2,5-di-(N-phenylalanino)-3,6-difluorquinone, 76, 123 - 124 and 179 - 180. In reaction of VII with amines and esters of amino acids a total substitution of F atom occurs with formation of corresponding (same data are presented): 2,5 diethyl-enimino-3,6-dipiperidinoquinone, 84, 175 - 176; diethyl ester of 2,5-diethylenimino-3,6-dipiperidinoquinone, 84, 175 - 176; diethyl ester of 2,5-diethylenimino-3,6-di-(N-alanino)-quinone, 25 - 30, 147.5 - 148; diethyl ester of 2, 5-diethylenimino-3.6-di(N-phenylalanino)-quinone, 20, 178 - 179. A synthesis of diethyleniminoquinones with amid groups was accomplished. For this by heating 2,5-dichloracetamino-3,6-dichlorquinone (VIII) with NH_3 in dioxane there was obtained 2,5-diglycylamino-3,6-dichlorquinone (IX), with yield of 85%, decomposition temperature $>360^\circ$ C. The heating of IX in medium VI led to 2,5-diglycylamino, 3,6-diethylenimino-quinone (X), yield 65%, temp. variable $>360^\circ$C. In the actions on X HCl (concentrate) there occurs a fractionizing of heterocycles with formation of chlorhydrates of 2.5-diglycylamino-3.6 di (β-chlorethylamino)-quinone, yield 65%, decomposition temperature $>360^\circ$C. In the action of VI on solutions VIII in dioxane was obtained 2.5-di-(ethyleniminoacetamino)-3.6-dichlorquinone (XI), yield 75%, m.p. 197°C (decomp.). The treatment of VII or XI with excess VI led to

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Substitution reactions in ....

a complete replacement of the Cl atoms with formation of 2,5-di-(ethyleniminoacetamino)-3,6-diethyleniminoquinone (XII), yield 80%, m.p. 217°C (decomp.). Under the action of HCl or HCl gas on XII or XI, corresponding /3-chlorethylamines were obtained. On the basis of the data obtained a series of replacements in the nucleus of benzoquinone were established in order of case when treated with amines or esters of amino acids. A series of synthesized substances were forwarded for oncological testing. S. Suminov.

/Abstractor's note: Complete translation/

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