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Nitration products of starch. I. Preparation of starch nitrates from potato starch. JULJUSZ HACKEL AND TADEUSZ URBAŃSKI. *Roczniki Chem.* 12, 276-97(in French 296-7)(1932).—In nitrating potato starch with HNO_3 the following conclusions are drawn: An increase of the acid concn. causes an increase of the N content and of the viscosity of the nitrates. Stabilizing boiling of the nitrates has a detrimental effect inducing denitration and an increase of the soly. in EtOH. The N content increases with increasing amt. of HNO_3 used. A rise of the temp. from 0° causes a lowering of the N content, of the yield and of the viscosity of the nitrates, accompanied by an increase of the EtOH soly. The changes are probably due to secondary oxidation reactions induced by the rise in temp. With increasing time of esterification the N content of the product rises rapidly at first, and then the increase becomes steadily smaller. The increase of the nitration period favors also the above secondary reactions, causing a drop of the yield, of the N content and of the viscosity of the product. Starch nitrates pptd. by H_2O show a better chem. stability than those pptd. by H_2SO_4. In nitrating starch with HNO_3-H_2SO_4-H_2O mixts. the best yield is obtained with mixts. containing equimolar amts. of H_2SO_4 and H_2O. Such mixts. showing a max. partial pressure of HNO_3 vapors exhibit a max. nitrifying power, and behave here in the same manner as in the nitration of cellulose. Mixts. poor in H_2SO_4 differ in their effect on starch from that on cellulose insofar as nitrated starch is probably sol. in them.

J. WIERTELAK

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Photographic studies of detonation of explosives. Tadeusz Urbański. *Roczniki Chem.* 12, 715-69(768-9 in French)(1932); cf. *C. A.* 27, 4930.—The detonation process of *picric acid* and *nitrocellulose* was studied by means of a high-speed camera permitting the dissection of a detonation flame into a series of pictures differing from each other by a distance of $^1/_{n,nn}$ of a sec. With picric acid the primary flame represents the main and partly the secondary reaction and consists of a center surrounded by luminous bundles. The progress of the secondary reaction depends on the rate of cooling of the reaction products effected by reduction of the pressure. The luminescence is due to glowing of free C and is induced by various reactions in the explosion space. The photo-graphic record permits the establishment of the following scheme of the explosion progress: The charge, being the center of the primary flame, is the place of the decompn. of the explosive. The decompn. products interact immediately, forming brighter reaction zones upon a darker field. This state is especially well perceived in photographing blue rays, in case the explosive is confined only with celluloid or is entirely free. If it is placed in a glass tube, the different zones run into each other causing brightening of the whole field, upon which darker bands are visible. The interacting decompn. products diverge and form luminous bands around the center. The glowing ceases with time toward the inner part whereby rings around the center are formed. The diam. of the rings increases steadily until the light is extinguished. The space between the center and the rings is illuminated by numerous radiant points. Analysis of the primary flame permits the conclusion that the reactions of the primary decompn. products do not proceed evenly in the whole mass, but are broken up into secondary centers forming rings around the place of the detonation. The flame along the charge axis shows all characteristics of an explosion flame. After extinction of the primary flame, a secondary one appears induced by oxidation of the reaction products: C, CO, CH_4, H_2, HCN, etc. The primary flame contains all colors of the visible spectrum; in the secondary one, blue is observed feebly, red and infra-red intensely. The lowest temp. limit of the primary flame is calcd. to 2000-2200°. Nitrocellulose behaves similarly, except that the form of the rings around the center is more regular, and their diam. is smaller than with picric acid. The lowest temp. of nitrocellulose, having 13.3% N, is calcd. to 1900-2000°. J. W.

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A-1

Phenomena associated with detonation of explosives. T. Urbański (Rocz. Chem., 1933, 13, [illegible]).—Mixtures of NH_4NO_3 with $C_6H_2Me(NO_2)_3$ or with glyceryl nitrate yield on detonation in glass tubes spirally propagated explosion flames. The velocity of propagation of the flame varies at different parts of the tube at the same distance from the origin. These effects are ascribed to asymmetrical action of the detonator, and to heterogeneity of the medium. A thin layer of explosive in contact with the walls of the tube may explode after the central mass, or may even remain intact after the explosion.

R. T.

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Nitration products of starch. II. Preparation of starch nitrates from starch from different sources. Juljusz Hackel and Tadeusz Urbański. *Roczniki Chem.* 13, 221-3(225 in French)(1933); cf. C. A. 27, [illegible].—No marked differences were found in the N content of nitrates prepared from corn, rice, tapioca or potato starch, or from sol. starch, in contradiction of the findings of Berl and Butler (C. A. 4, 1550).

T. H. Chilton

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CA

Thermal analysis of binary mixtures containing organic nitrates. Tadeusz Urbański. *Roczniki Chem.* 13, 399-434(1933).—A 2.5-g. sample, in a 12-mm. tube placed in a 16-mm. tube which in turn is placed in a 35-mm. tube contg. glycerol, is heated to 5-10° above its m. p. During the cooling, the melt and glycerol are stirred vigorously. Temp.-compn. curves are given. The following figures are, resp., the percentage of the 1st ingredient and the eutectic pt. Mannitol nitrate-*p*-nitrotoluene, 12.5, [illegible]9.2°; -*p*-nitroanisole 8, 50.8°; -*p*-nitrophenetole, 5, [illegible].8°; -*o*-chloronitrobenzene, 15.5, 29.7°; -α-nitronaphthalene, 14, 52.5°; -*m*-dinitrobenzene, 52.5, 65.5°; -1,[illegible]-dinitroanisole, 55, 77.6°; -1,2,4-chlorodinitrobenzene, 25, 44.5°; -*sym*-trinitrobenzene, 55, 78.7°; -α-trinitrotoluene, 42.5, 62.8°; -naphthalene, 20, 77.3°; -dimethyldiphenylurea, 57, 77.4°; -diethyldiphenylurea, 53-4, approx. 82.1°. Nitroerythritol-*p*-nitrotoluene, 47, 32.4°; -*m*-dinitrobenzene, 70, 42.4°. Pentaerythritol nitrate-*p*-nitrotoluene, 10, 50.2°; -*p*-nitroanisole, 5, 50.8°; -α-nitronaphthalene, 4, 55.1°; -*m*-dinitrobenzene, 20, 82.4°; -1,3,5-dinitroanisole, 20, 94.7°; -1,2,4-chlorodinitrobenzene, 3, 49°; -*sym*-trinitrobenzene, approx. 30, 101.1°; -α-trinitrotoluene, approx. 13, 76.1°; -naphthalene, approx. 7, 79.1°; -dimethyldiphenylurea, 32.5, 102.7°; -diethyldiphenylurea, approx. 12, 68°; mannitol nitrate-erythritol nitrate 18.5, 57.6°; pentaerythritol nitrate-mannitol nitrate, 20, 101.3°; pentaerythritol nitrate-erythritol nitrate, approx 5, 59.5°. Mannitol nitrate was the only compd. giving rise to mol. addn. compds. Probable compns. (detd. by extrapolation) are, in mols. per mol. of mannitol nitrate, *p*-nitrotoluene 2, *p*-nitroanisole 2, *p*-nitrophenetole 2, α-nitronaphthalene. 1. Bibliography. C. T. I.

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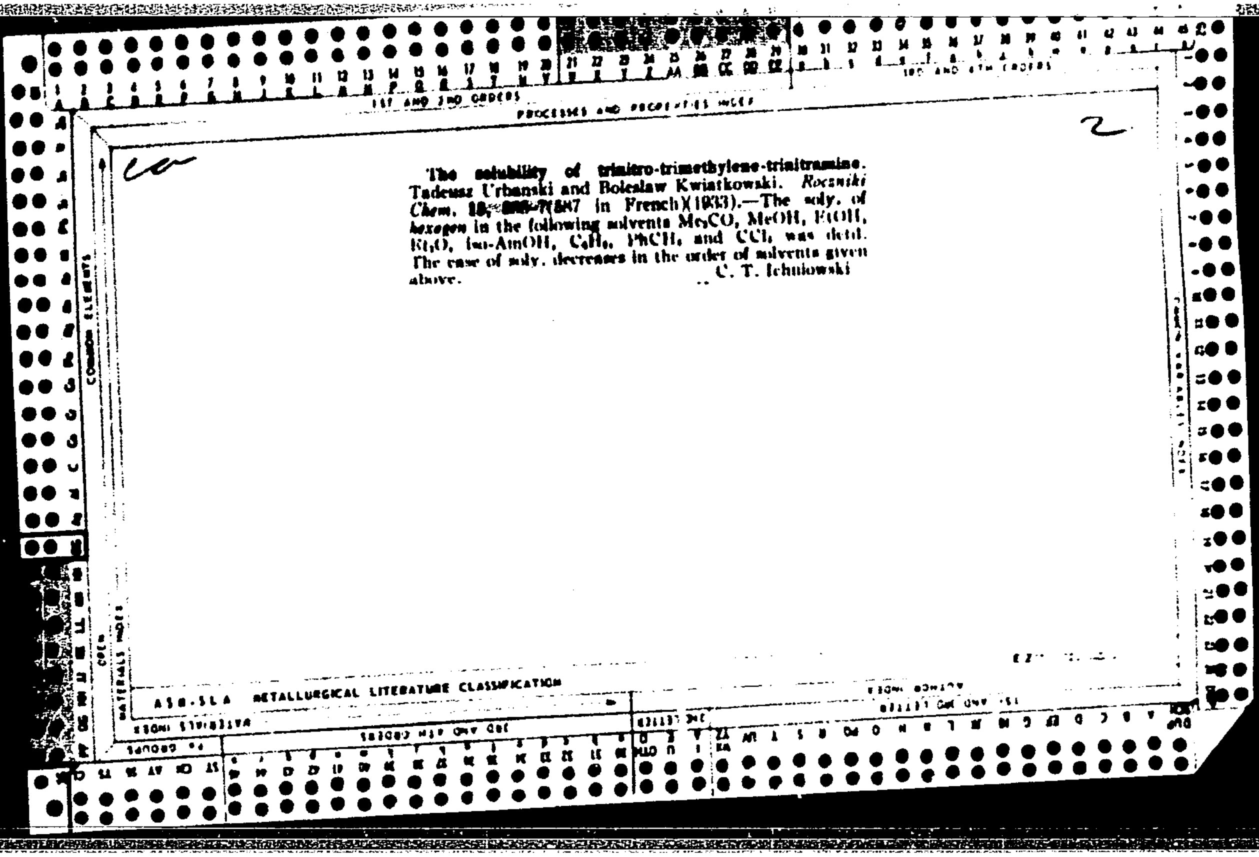

ca

2

The solubility of trinitro-trimethylene-trinitramine. Tadeusz Urbanski and Boleslaw Kwiatkowski. *Roczniki Chem.* 13, [illegible]-7(587 in French)(1933).—The soly. of *hexogen* in the following solvents Me_2CO, MeOH, EtOH, Et_2O, iso-AmOH, C_6H_6, $PhCH_3$, and CCl_4 was detd. The ease of soly. decreases in the order of solvents given above.

C. T. Ichniowski

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Bc

4-1

CHEMICAL ELEMENTS

Solubility of [illegible] T.
U[illegible] 1953,
[illegible] iron,
H_2O, [illegible] and
the b.p. [illegible] R. T.

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Thermal analysis of mixtures containing trinitrotrimethylenetriamine. T. Urbański and I. Kapuścińska-Gawrońska (Rocz. Chem., [illegible]—245).—Neither solutions nor compounds are formed in the systems trinitrotrimethylenetriamine—p-$C_6H_4Me{\cdot}NO_2$, p-$NO_2{\cdot}C_6H_4{\cdot}OMe$, 1-$C_{10}H_7{\cdot}NO_2$, m-$C_6H_4(NO_2)_2$, s-$C_6H_3(NO_2)_3$, $C_6H_2Me(NO_2)_3$, $C_6H_2(NO_2)_3{\cdot}OH$, tetryl, $NPh_2{\cdot}CO{\cdot}NEt_2$, $NPh_2{\cdot}CO{\cdot}NH_2$, camphor, and $NHPh_2$. R. T.

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Thermal analysis of binary mixtures containing organic nitrates. T. Urbański. *Roczniki Chem.* 14, 925-40 (1934); cf. *C. A.* 28, 27^1.—The m.-p. diagrams of the systems mannitol hexanitrate (I)-*p*-NO$_2$C$_6$H$_4$OH (II), *p*-C$_6$H$_4$C[illegible]NO$_2$, *p*-C$_6$H$_4$(OMe)$_2$, *m*-(III) and *p*-(IV)NO$_2$C$_6$H$_4$CO$_2$Et; *m*-(V) and *p*-NO$_2$C$_6$H$_4$CHO, *m*-NH$_2$C$_6$H$_4$NO$_2$ and 1,2,4-C$_6$H$_3$Me(NO$_2$)$_2$ (VI); erythritol tetranitrate (VII)-([illegible]) and 2,4,6-trinitrotoluene (VIII); pentaerythritol tetranitrate(IX)-(VI), trinitrophenylmethylnitroaniline, [illegible]NH$_2$Ph, and camphor (X), indicate the unstable compds. 2I.III, 2I.IV, I.2V and VII.4VIII. Solid solns. are formed in the system IX X, while in the remaining systems only ordinary eutectic mixts. are observed.

B. C. A.

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Thermal analysis of the system picric acid-dinitronaphthalene. T. Urbański and B. Kwiatkowski. *Roczniki Chem.* 14, 941-6(1934).—Neither 1,5- nor 1,8-$C_{10}H_6(NO_2)_2$ gives rise to compds. or solid solns.

B. C. A.

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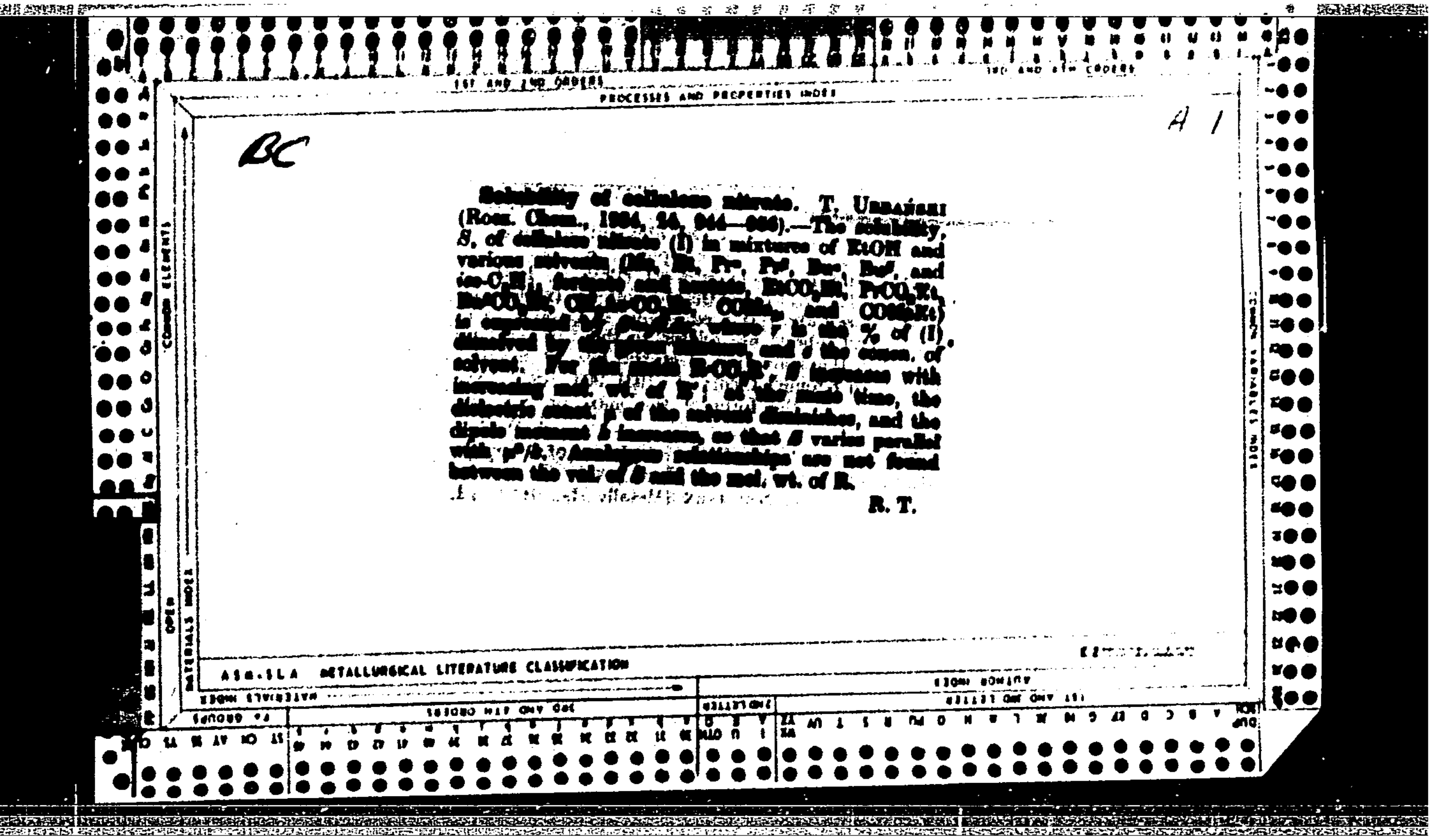
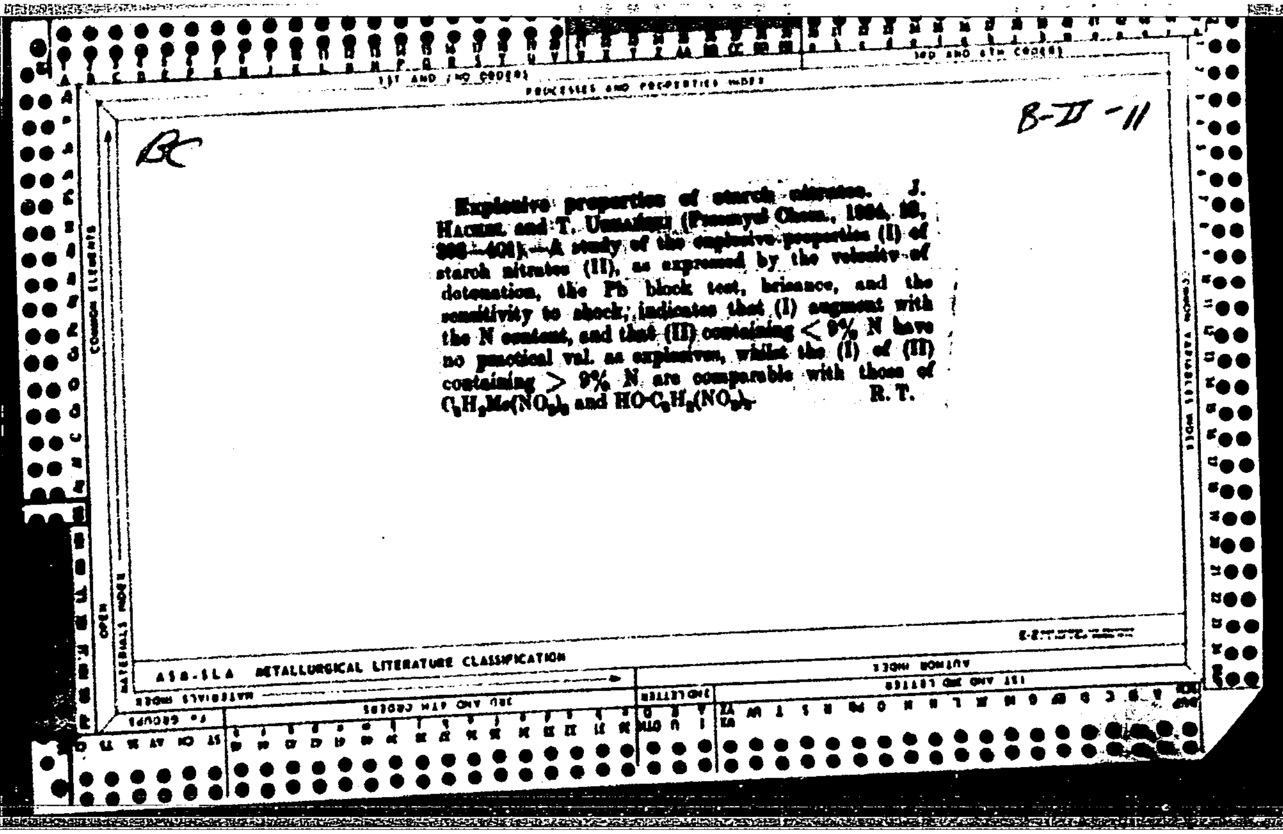


Solubility of cellulose nitrate. T. Urbański (Rocz. Chem., 1954, 28, 944—956).—The solubility, *S*, of cellulose nitrate (I) in mixtures of EtOH and various solvents (Me, Et, Prα, Prβ, Buα, Buβ, and iso-C_5H_{11} formates and acetates, $EtCO_2Et$, $PrCO_2Et$, $BuCO_2Et$, $CH_2Cl \cdot CO_2Et$, $CO(OMe)_2$, and $CO(OEt)_2$) is expressed by $S = p/c$, where *p* is the % of (I) dissolved by the given mixture, and *c* the concn. of solvent. For the esters $R \cdot CO_2R'$, *S* increases with increasing mol. wt. of R'; at the same time, the dielectric const. ε of the solvent diminishes, and the dipole moment μ increases, so that *S* varies parallel with μ^2/ε. Analogous relationships are not found between the val. of *S* and the mol. wt. of R.

R. T.



BC

B-II-11

Explosive properties of starch nitrates. J. Hackel and T. Urbański (Przemysł Chem., 1934, 18, 398–401).—A study of the explosive properties (I) of starch nitrates (II), as expressed by the velocity of detonation, the Pb block test, brisance, and the sensitivity to shock, indicates that (I) augment with the N content, and that (II) containing < 9% N have no practical val. as explosives, whilst the (I) of (II) containing > 9% N are comparable with those of $C_6H_2Me(NO_2)_3$ and $HO·C_6H_2(NO_2)_3$. R. T.

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Thermal analysis of binary mixtures containing organic trates. III. T. Urbanski. *Roczniki Chem.* 15, 191-7 935); cf. *C. A.* 29, 6129^{4}.—Temp.-compn. curves of e systems studied are given. The accompanying figures re resp. the percentage of the first ingredient and the tectic point. Mannitol nitrate-*m*-nitrotoluene forms n addn. compd. (in ratio of 1:2) which forms a eutectic ith mannitol nitrate, 8, 14.4°; mannitol nitrate-amphor, 45, 5.8°. Erythritol nitrate-1,2,4-dinitrotolune, 61, 40.1°; -*sym.*-trinitrobenzene, 67, 45.8°; -diethyliphenylurea, 37, 42.2°; erythritol pentanitrate-phthalde, 20, 65.9°; -Et phenylcarbamate, 1, 48.6°; erythritol itrate-1,2,4,6-trinitroanisole forms an addn. compd. 1 + 2) and yields 2 eutectics: one with the nitroanisole and the other with erythritol nitrate m. at 58.6° and 52° and corresponding to 10 and 70% of erythritol nitrate, resp.

C. T. Ichniowski

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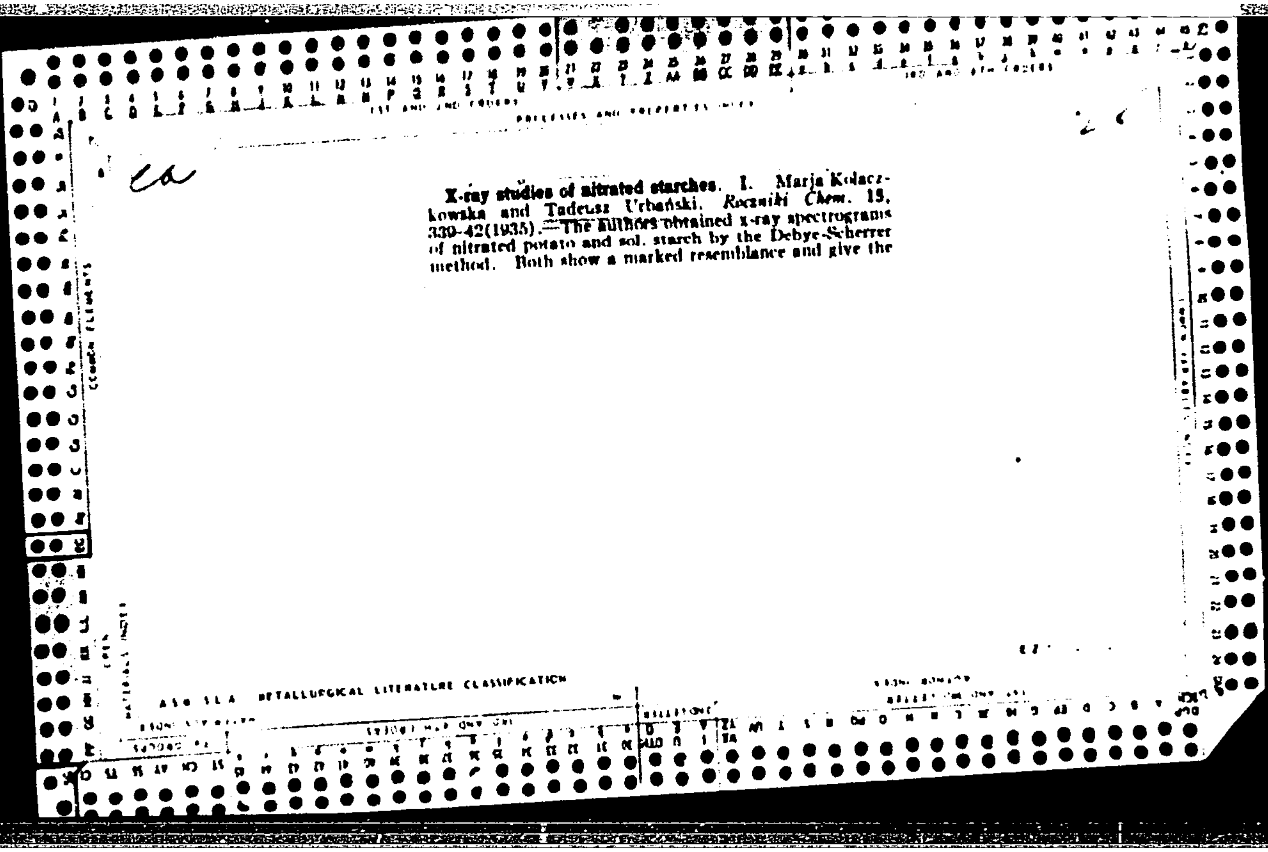

ca

X-ray studies of nitrated starches. I. Marja Kołaczkowska and Tadeusz Urbański. *Roczniki Chem.* 15, 330-42(1935).—The authors obtained x-ray spectrograms of nitrated potato and sol. starch by the Debye-Scherrer method. Both show a marked resemblance and give the

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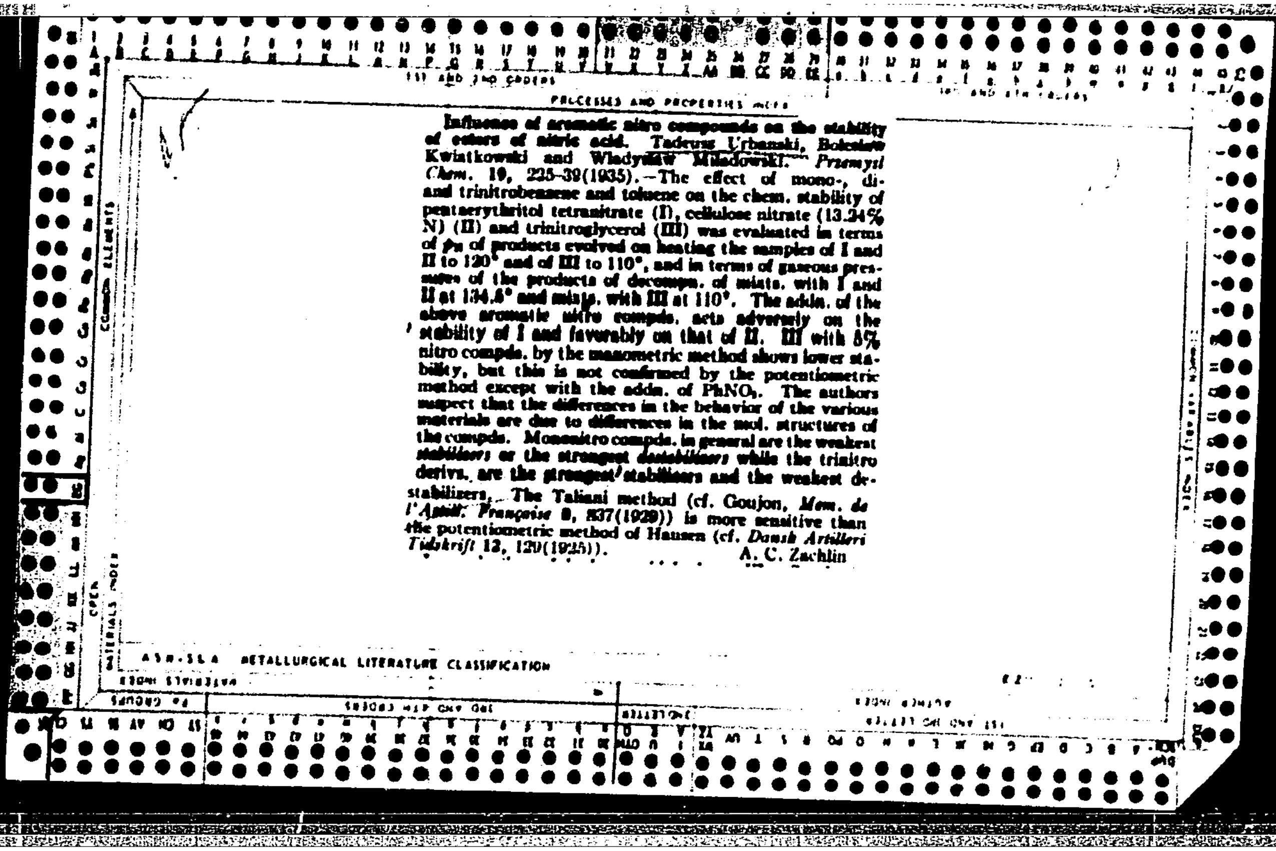

Influence of aromatic nitro compounds on the stability of esters of nitric acid. Tadeusz Urbanski, Boleslaw Kwiatkowski and Wladyslaw Miladowski. *Przemysl Chem.* 19, 225-39(1935).—The effect of mono-, di- and trinitrobenzene and toluene on the chem. stability of pentaerythritol tetranitrate (I), cellulose nitrate (13.24% N) (II) and trinitroglycerol (III) was evaluated in terms of pH of products evolved on heating the samples of I and II to 120° and of III to 110°, and in terms of gaseous pressures of the products of decompn. of mixts. with I and II at 134.5° and mixts. with III at 110°. The addn. of the above aromatic nitro compds. acts adversely on the stability of I and favorably on that of II. III with 5% nitro compds. by the manometric method shows lower stability, but this is not confirmed by the potentiometric method except with the addn. of $PhNO_2$. The authors suspect that the differences in the behavior of the various materials are due to differences in the mol. structures of the compds. Mononitro compds. in general are the weakest *stabilizers* or the strongest *destabilizers* while the trinitro derivs. are the strongest stabilizers and the weakest destabilizers. The Taliani method (cf. Goujon, *Mem. de l'Artill. Française* 8, 837(1929)) is more sensitive than the potentiometric method of Hansen (cf. *Dansk Artilleri Tidsskrift* 12, 120(1925)).

A. C. Zachlin

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NN'-Dimethyldiamide of tartaric acid and the *NN'*-dinitrodimethyldiamide of tartaric acid dinitrate. T. URBAŃSKI (Rocz. Chem., 1936, 16, 224—230).—The velocity of reaction between NH_2Me and esters of tartaric acid, and the yield of *NN'*-dimethyldiamide (I) of tartaric acid, m.p. 212—214° (lit., 189°), fall in the series Me > Et > Pr^{β} tartrate. *Tartaric acid dinitrate NN'-dinitrodimethyldiamide* (II), m.p. 116° (decomp.), is obtained by adding 60 g. of Ac_2O to 10 g. of (I) in 100 g. of HNO_3 at > −2°. (II) is readily detonated by shock or heat, and yields gels with cellulose nitrate. R. T.

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a-1

Thermal analysis of mixtures of ammonium nitrate, guanidine nitrate, and nitroguanidine. T. U[illegible] and J. [illegible] (Rocz. Chem., [illegible], 16, [illegible]).—Data for the binary eutectics are: NH_4NO_3 (I) 74, guanidine nitrate (II) 26%, f.p. 127·7°; (I) [illegible], nitroguanidine (III) 20%, f.p. 131·5°, (II) [illegible], (III) 41%, f.p. [illegible]; those for the ternary eutectic are; (I) [illegible], (II) 22·5, (III) 17·5%, f.p. 113·2°. R. T.

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Thermal analysis of binary mixtures containing esters of nitric acid. IV. T. Urbański (Rocz. Chem., 1936, 16, [illegible]—365).—The data suggest formation of unstable 1 : 2 compounds of mannitol hexanitrate (I) with PhNO$_2$, o-C$_6$H$_4$Me·NO$_2$, and Et o-nitrocinnamate, and of a 2 : 1 compound with Et o-nitrobenzoate. Simple eutectics are formed in the systems: (I)–NHPh·CO$_2$Et, erythritol tetranitrate–s-OEt·C$_6$H$_3$(NO$_2$)$_2$, and pentaerythritol tetranitrate–m-NO$_2$·C$_6$H$_4$·NH$_2$ and -Me$_2$C$_2$O$_4$. R. T.

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Thermal analysis of mixtures containing ammonium, sodium, potassium, and calcium nitrates. T. Urbański and S. Kołodziejczyk (Rocz. Chem., 1936, 16, 119—125).—The system NH_4NO_3-$Ca(NO_3)_2$ (eutectic, 71% of NH_4NO_3, 111°) belongs to Rosenbaum's type V. Thermal diagrams for the systems NH_4NO_3-$NaNO_3$-KNO_3 and NH_4NO_3-$NaNO_3$-$Ca(NO_3)_2$ have been determined.

R. T.

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Susceptibility of explosives to shock. I. Method. Tadeusz Urbanski. *Przemysl Chem.* 20, 117-27(1936).—The literature is reviewed; 24 references are given. A modification of the methods of Lenze (*Atti VI Congr. Intern.* 2, 572(1907)) and Kast (*C. A.* 3, 2220) is adopted. Agreement between results can be improved if the following precautions are observed: The plunger of the app. should be subjected to more than 10 or 20 strokes before making the detns. in order to arrive at such a condition that the end of the plunger will not be further deformed by the blow. The harder the steel the fewer the blows needed to stabilize the plunger. U. shows clearly the effect of such stabilization. The rebound for various wts. falling from various heights should be detd. for each lot of plungers similarly prepd. from the same material. From this the work done by the wt. is calcd. and expressed in kg./sq. cm. At least two app. should be used alternately. The quantity of substance used each time should be standardized at 0.01 to 0.02 g. The substance should be pulverized to a grain size of about 0.1 mm. diam. The app. should be cleaned after every stroke, and all deformations polished away. In order sufficiently to describe the behavior of the material under shock at least 2 heights should be detd. with an accuracy of 1.0 cm.: the min. at which 10% explosions are obtained and the max. for 50% of explosions. Each of the above should be detd. on the basis of at least 10 readings. In view of the varied behaviors of different explosives under shock at least 2 standard substances should be used, e. g., *hexogen* for the more sensitive substances and *picric acid* for the less sensitive substances. When the report of the explosion is questionable, then the appearance of soot or the presence of odor of combustion should serve as addnl. criteria of explosion. An accuracy of 10% is obtained. **II. Sensibility of mixtures.** *Ibid.* 179-87.—By the method described above, mixts. of *penthrite* and *hexogen* with various nitro compds., an O carrier, and a neutral salt were studied. Mixts. of 2 explosive substances of different sensibilities show a sensibility which varies with compn., but the transition of mixts. from those rich in the sensitive component to those rich in the other shows a pronounced discontinuity. If a not very sensitive substance, e. g., *tetryl*, *trotyl* or *picric acid*, is added to the extent of 10% to a sensitive substance such as *hexogen* or *penthryl* the sensitiveness of the latter is increased. Sensitiveness is also increased by the addn. of an O carrier such as KNO_3 up to 60% or of a neutral salt (KCl). The addn. of a small quantity (5%) of a sensitive substance such as hexogen or penthryl to such substances as nitro compds. increases the sensitiveness markedly. The above observations are interpreted to point to one conclusion: the increase in sensitiveness is produced by mech. sepn. of crystals of the foreign substance, which sepn. results in greater interior friction of the mass. A. C. Z.

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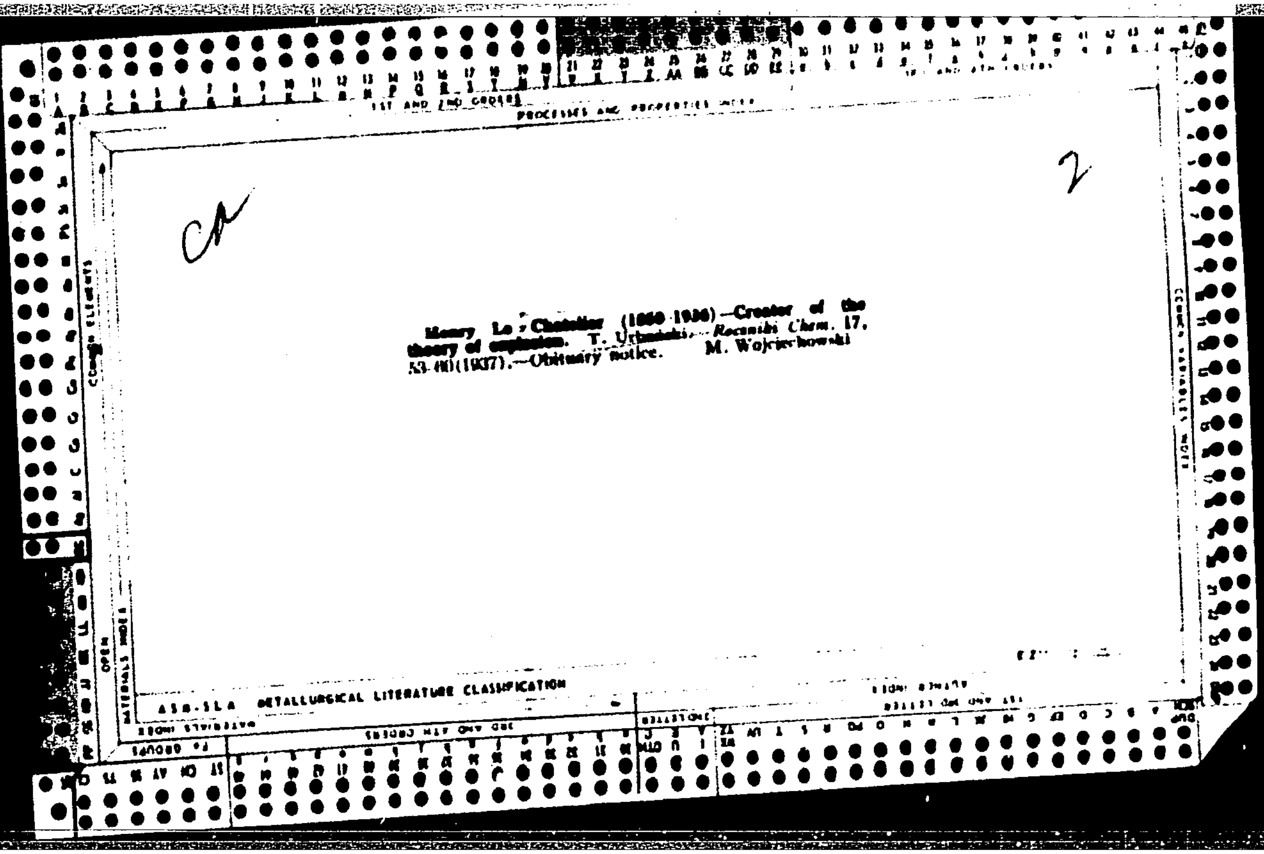

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Henry Le Chatelier (1850-1936)—Creator of the theory of explosion. T. Urbanski.—Roczniki Chem. 17, 53-60(1937).—Obituary notice. M. Wojciechowski

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Nitration of paraffin hydrocarbons. II. T. Urbanski and M. Slon. *Roczniki Chem.* 17, 161-4 (in French 164) (1937); cf. *C. A.* 31, 654^7.—Continuing the study of nitration of paraffin hydrocarbons with NO_2 in the gaseous phase, hydrocarbons from pentane to nonane were nitrated. The process was conducted at 200° and a mixt. of mono- and di-nitro compds. has been obtained with an efficiency of 30-80%, depending on the conditions of reaction. The formulas of the products are: $O_2NCH_2(CH_2)_nMe$ and $O_2NCH_2(CH_2)_nCH_2NO_2$. M. Wojciechowski

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Nitration of cellulose and starch by means of nitric anhydride. T. Urbański and Z. Janiszewski. *Roczniki Chem.* 17, 249-52(in French 252)(1937).—The authors have examd. the nitrating action of N_2O_5 on cellulose and starch. By the action of N_2O_5 it is possible to obtain nitrocellulose contg. about 14% of N. The same result is obtained when liquid N_2O_4 mixed with HNO_3 acts on cellulose. Starch nitrated with gaseous or liquid N_2O_5 contains up to 13.86% of N. M. Wojciechowski

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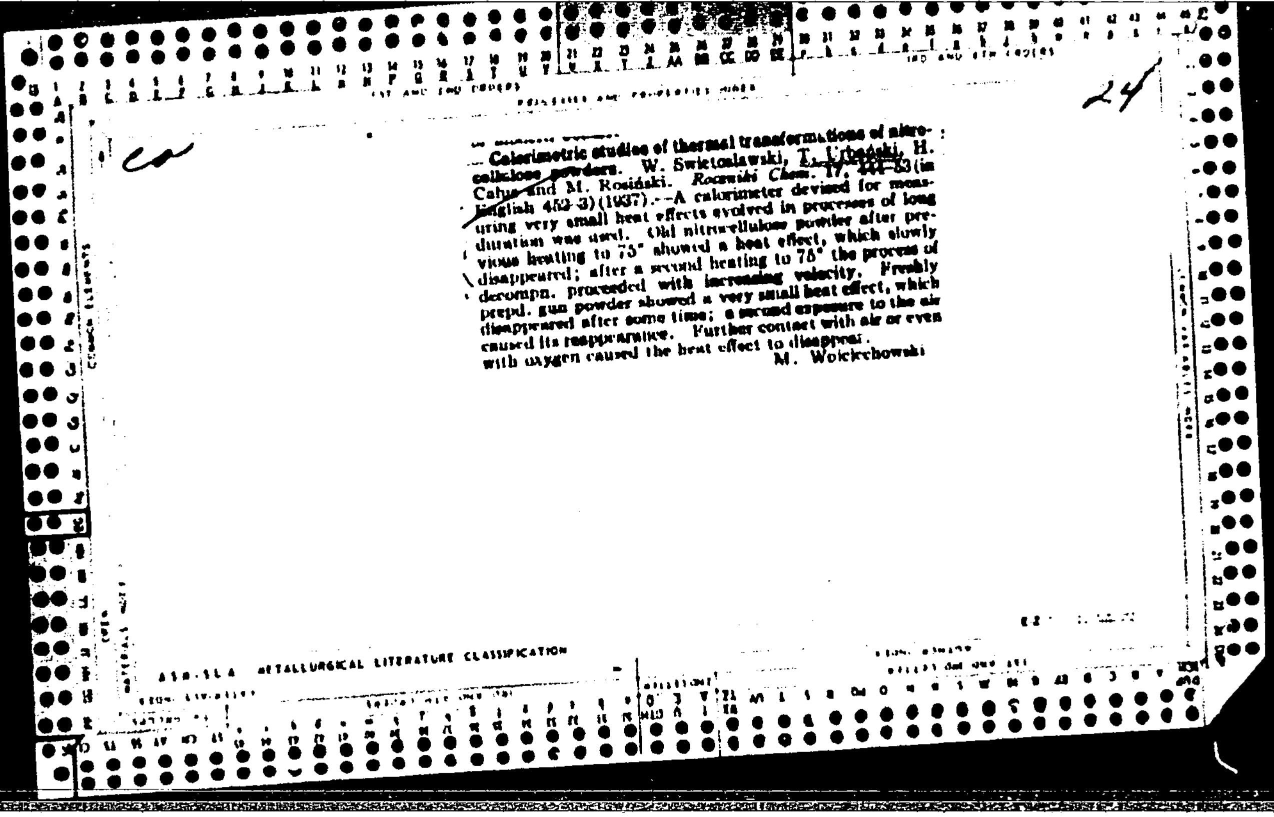

ca

24

Calorimetric studies of thermal transformations of nitrocellulose powders. W. Swietoslawski, T. Urbański, H. Calus and M. Rosiński. *Roczniki Chem.* 17, 444-53 (in English 452-3) (1937).--A calorimeter devised for measuring very small heat effects evolved in processes of long duration was used. Old nitrocellulose powder after previous heating to 75° showed a heat effect, which slowly disappeared; after a second heating to 75° the process of decompn. proceeded with increasing velocity. Freshly prepd. gun powder showed a very small heat effect, which disappeared after some time; a second exposure to the air caused its reappearance. Further contact with air or even with oxygen caused the heat effect to disappear.

M. Wojciechowski

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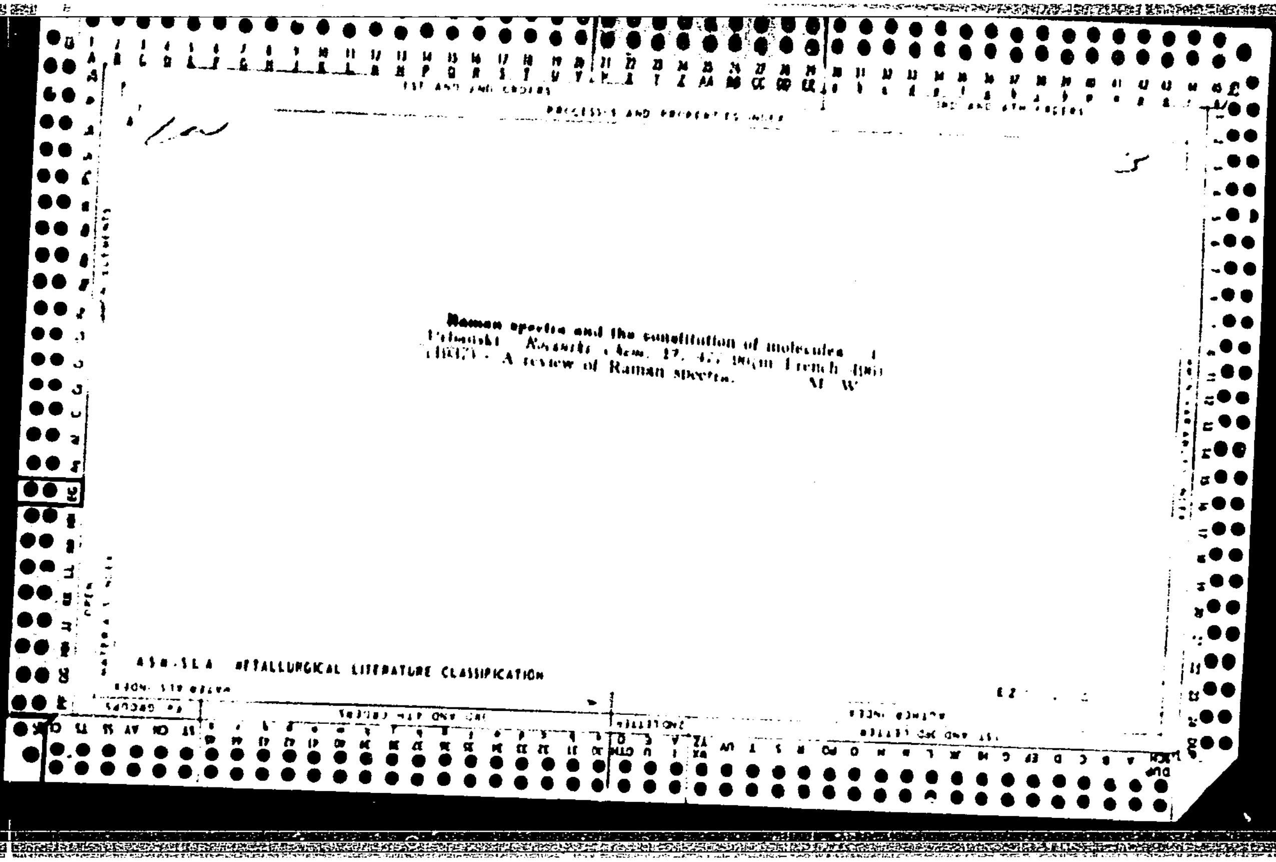

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3RD AND 4TH ORDERS

Raman spectra and the constitution of molecules I. [illegible]. Roczniki Chem. 17, 45, 90(in French) [illegible] (1937). - A review of Raman spectra. M. W.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

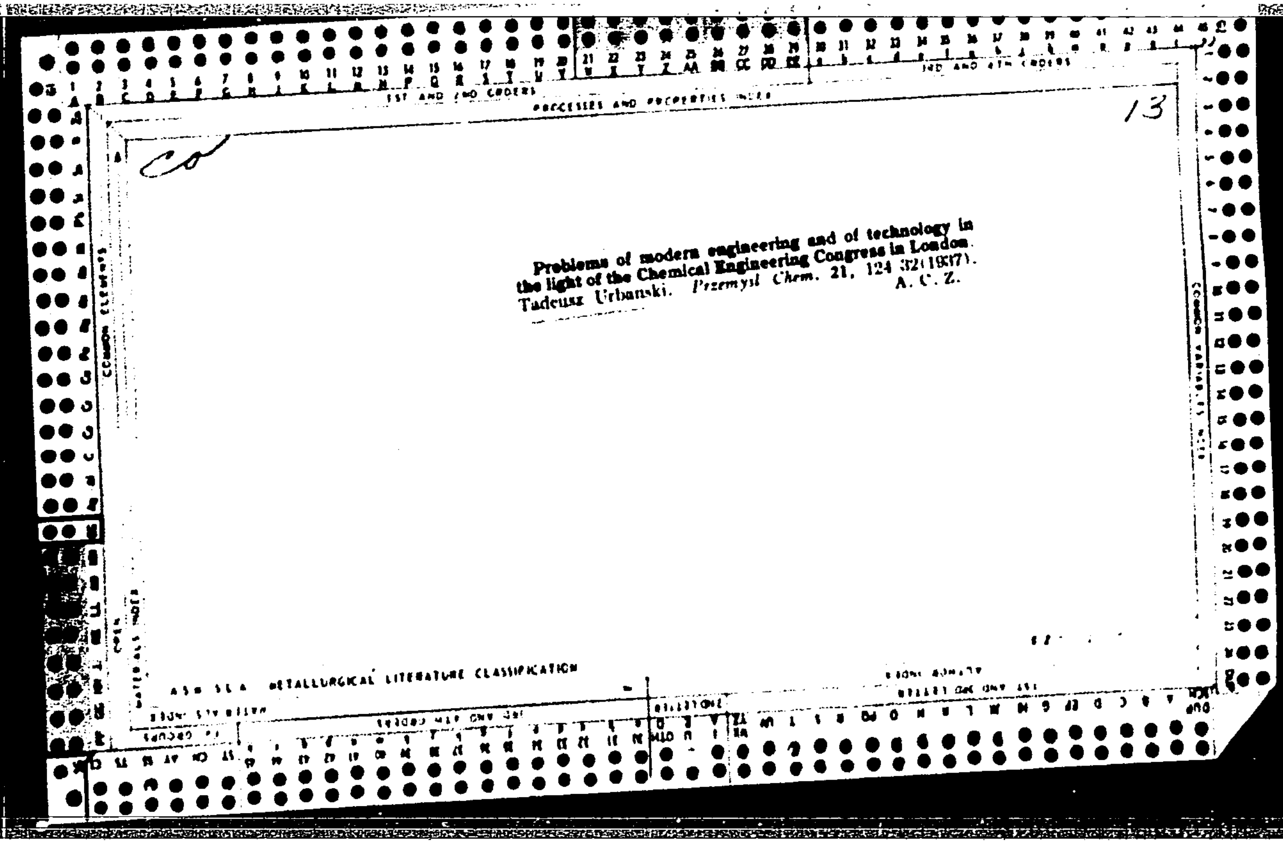
PROCESSES AND PROPERTIES INDEX

24

ca

Preparation of trinitrotris(methylamino)benzene. T. Urbański. *Roczniki Chem.* 17, 501-3 (in French 502-3) (1937).—U. has prepd. 2,4,6-trinitro-1,3,5-tris(methylamino)benzene (I) by boiling *s*-trinitrotrichlorobenzene with an aq. soln. of methylamine. I gives with concd. HNO_3 the nitroamine, 2,4,6-trinitro-1,3,5-tris(methylnitroamino)benzene, which is a very powerful explosive, stronger than tetryl. M. Wojciechowski

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION



Co

13

Problems of modern engineering and of technology in the light of the Chemical Engineering Congress in London. Tadeusz Urbanski. *Przemysl Chem.* 21, 124 32(1937). A. C. Z.

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Nitration of paraffin hydrocarbons. T. Urbański and M. Słoń. *Przeglad Chem.* 2, 42-3(1938).—Summarized report of the authors' expts. on the nitration of aliphatic hydrocarbons with NO_2 in the gaseous phase (cf. *C. A.* 31, 3868⁴, 6190⁴).

F. Józefowicz

COMMON ELEMENTS

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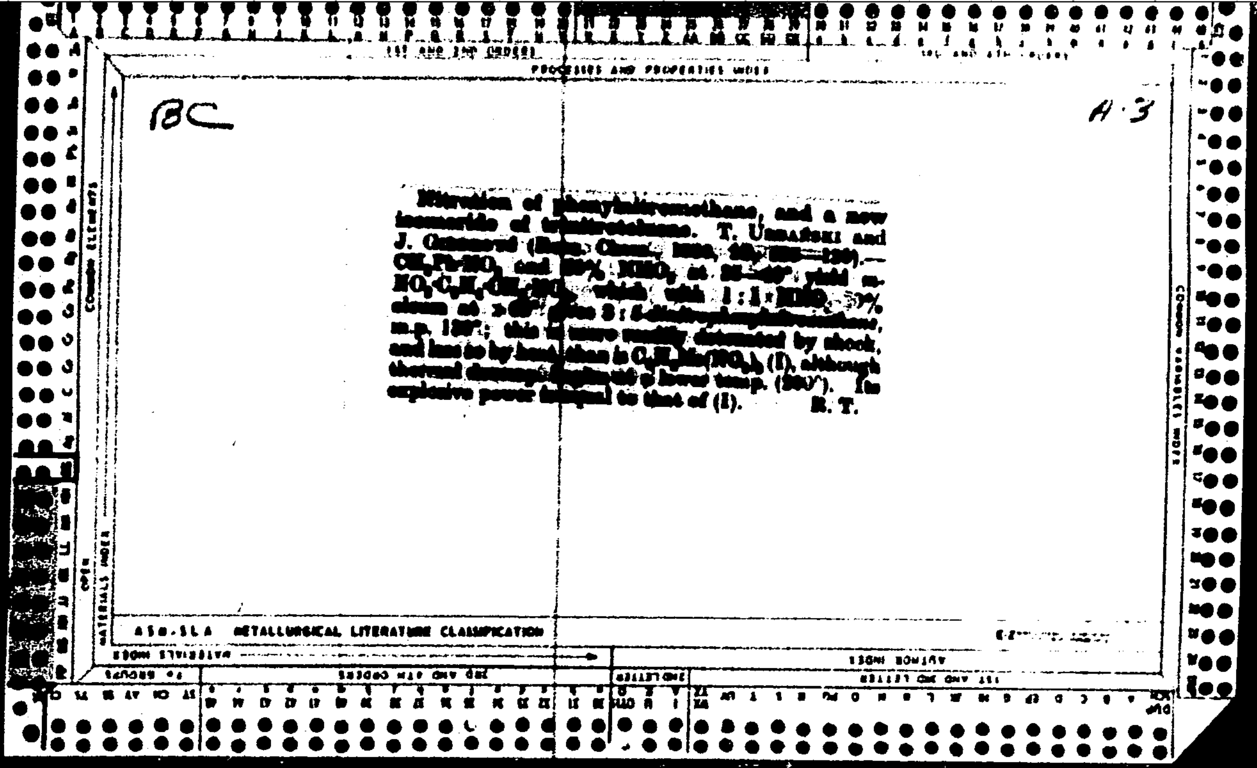
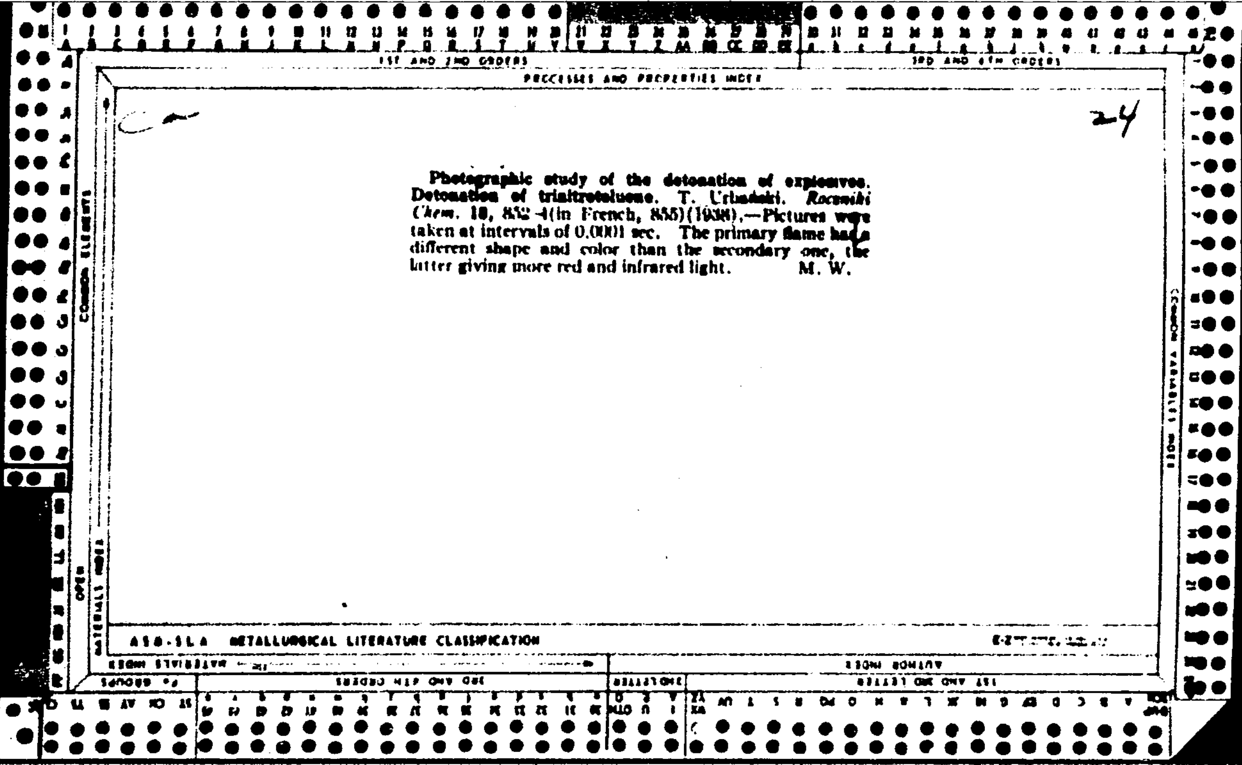


BC

A-3

Nitration of phenylnitromethane, and a new isomeride of trinitrotoluene. T. Urbański and J. Gurzyński (Rocz. Chem., [illegible], 18, [illegible]).—$CH_2Ph\cdot NO_2$ and 98% HNO_3 at 35—40° yield m-$NO_2\cdot C_6H_4\cdot CH_2\cdot NO_2$, which, with 1 : 1 HNO_3–37% oleum at >60° gives 3 : 5-dinitrophenylnitromethane, m.p. 120°; this is more readily detonated by shock, and less so by heat, than is $C_6H_2Me(NO_2)_3$ (I), although thermal decomposition sets in at a lower temp. (200°). Its explosive power is equal to that of (I). R. T.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION



1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

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24

Photographic study of the detonation of explosives. Detonation of trinitrotoluene. T. Urbański. *Roczniki Chem.* 18, 852-4 (in French, 855) (1938).—Pictures were taken at intervals of 0.0001 sec. The primary flame had a different shape and color than the secondary one, the latter giving more red and infrared light. M. W.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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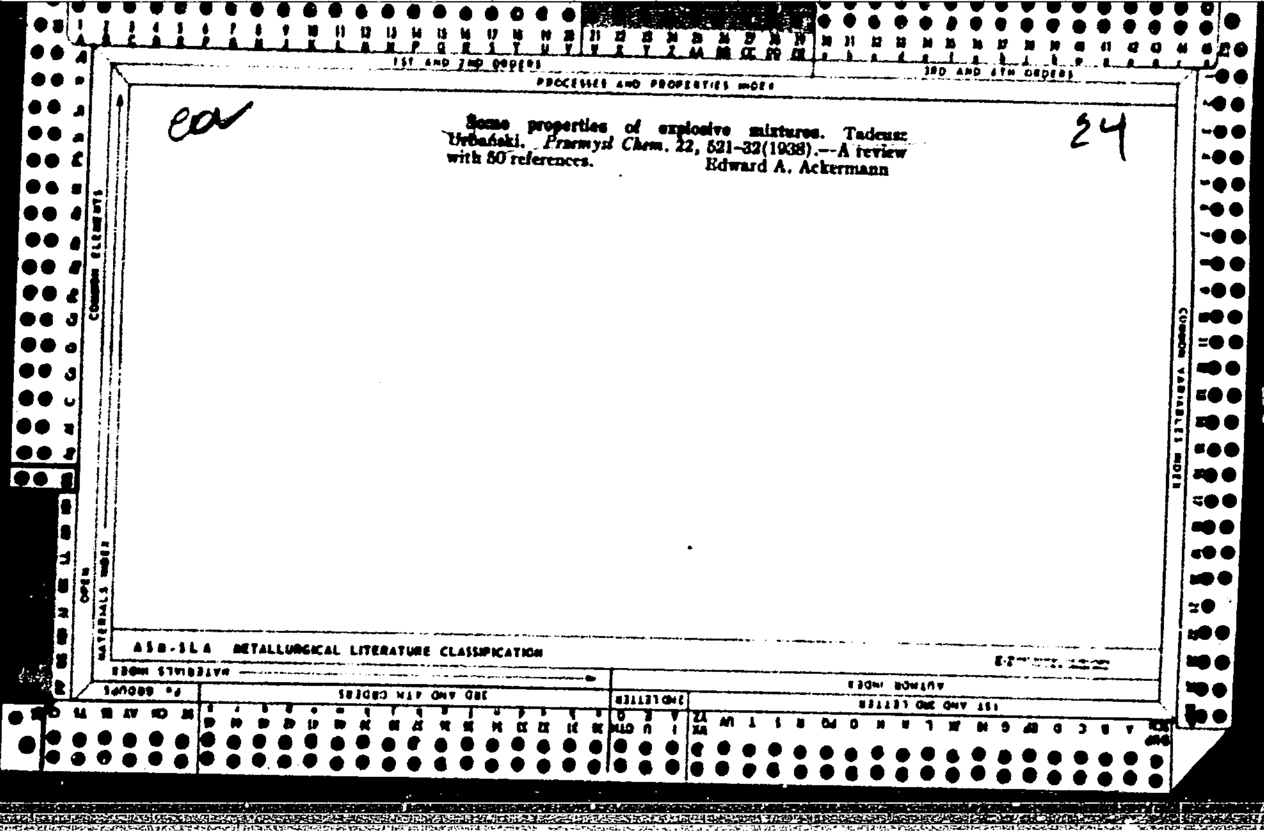
C

2

Influence of ultraviolet rays on a few explosives. T. Urbański and W. Malendowicz. *Roczniki Chem.* 18, 860-63 (in French, 863-4) (1938).—The influence of ultraviolet rays on nitroglycerin, nitropentaerythritol, hexogen, and nitrocellulose and nitroglycerin powders was studied. The decompn. took place with const. rate. The decompn. decreased in the order nitroglycerin, nitroerythritol, nitromannitol and nitrocellulose. In general, the smaller the mol. wt. of the compd. the higher the rate of decompn. The more nitro groups there are in a mol., the more rapidly does decompn. proceed. M. W.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION



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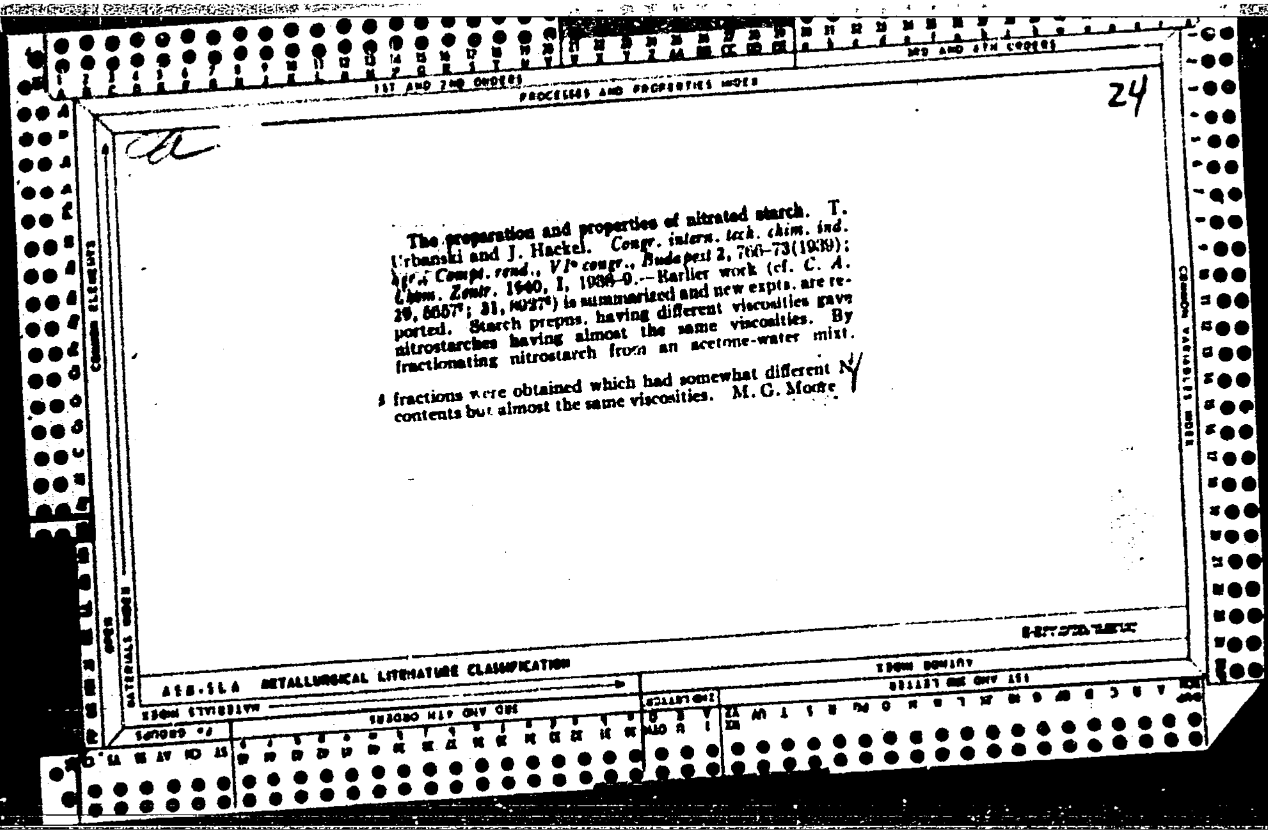
ed

24

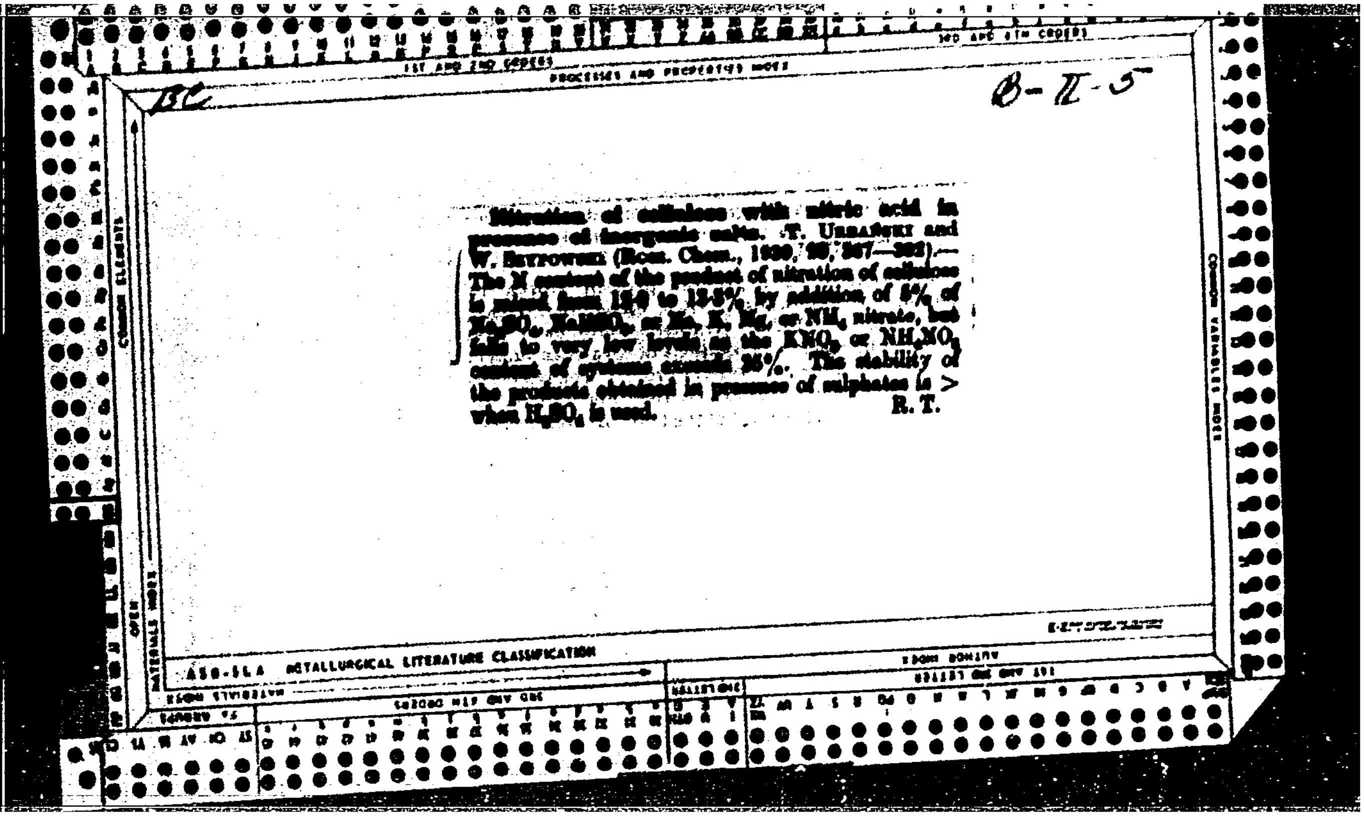
Some properties of explosive mixtures. Tadeusz Urbański. *Przemysl Chem.* 22, 521-32(1938).—A review with 50 references. Edward A. Ackermann

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION



24

The preparation and properties of nitrated starch. T. Urbanski and J. Hackel. *Congr. intern. tech. chim. ind. Agr., Compt. rend., VIe congr., Budapest* 2, 766-73(1939); *Chem. Zentr.* 1940, I, 1936-9.—Earlier work (cf. *C. A.* 29, 6657⁵; 31, 8027⁸) is summarized and new expts. are reported. Starch prepns. having different viscosities gave nitrostarches having almost the same viscosities. By fractionating nitrostarch from an acetone-water mixt. 4 fractions were obtained which had somewhat different N contents but almost the same viscosities. M. G. Moore



BC

B-II-5

Nitration of cellulose with nitric acid in presence of inorganic salts. T. Urbański and W. Sztypowski (Rocz. Chem., 1930, 10, 287—302).—The N content of the product of nitration of cellulose is raised from 13·0 to 13·5% by addition of 5% of Na_2SO_4, $NaHSO_4$, or Na, K, Mg, or NH_4 nitrate, but falls to very low levels as the KNO_3 or NH_4NO_3 content of systems exceeds 25%. The stability of the products obtained in presence of sulphates is > when H_2SO_4 is used.

R. T.

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

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CA

10

Possible dimorphism of trinitrobenzene. Tadeus Urbański and J. Simon. *Roczniki Chem.* 19, 487-91(1939).—Nitration of m-$C_6H_4(NO_2)_2$ with HNO_3 and 60% oleum gives 1,3,5-$C_6H_3(NO_2)_3$ (I), m. 121°, or a substance, m. 61-2°, presumably identical with that described by Radcliffe and Pollitt (*C. A.* 15, [illegible]) as being a polymorph of I. This product is shown to be a mixt. of m-$C_6H_4(NO_2)_2$ 35-50 and I 50-65%. B. C. P. A.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

New developments in the field of rubber vulcanization. T. Urbański. *Przemysł Chem.* 3, 231-3(1947)(in Polish). The possibility of vulcanizing rubber with phenol-formaldehyde resins is discussed on the basis of the work of Hultzsch, v. Euler, Cunneen, Farmer, Koch, and van der Meer. Frank Gonet

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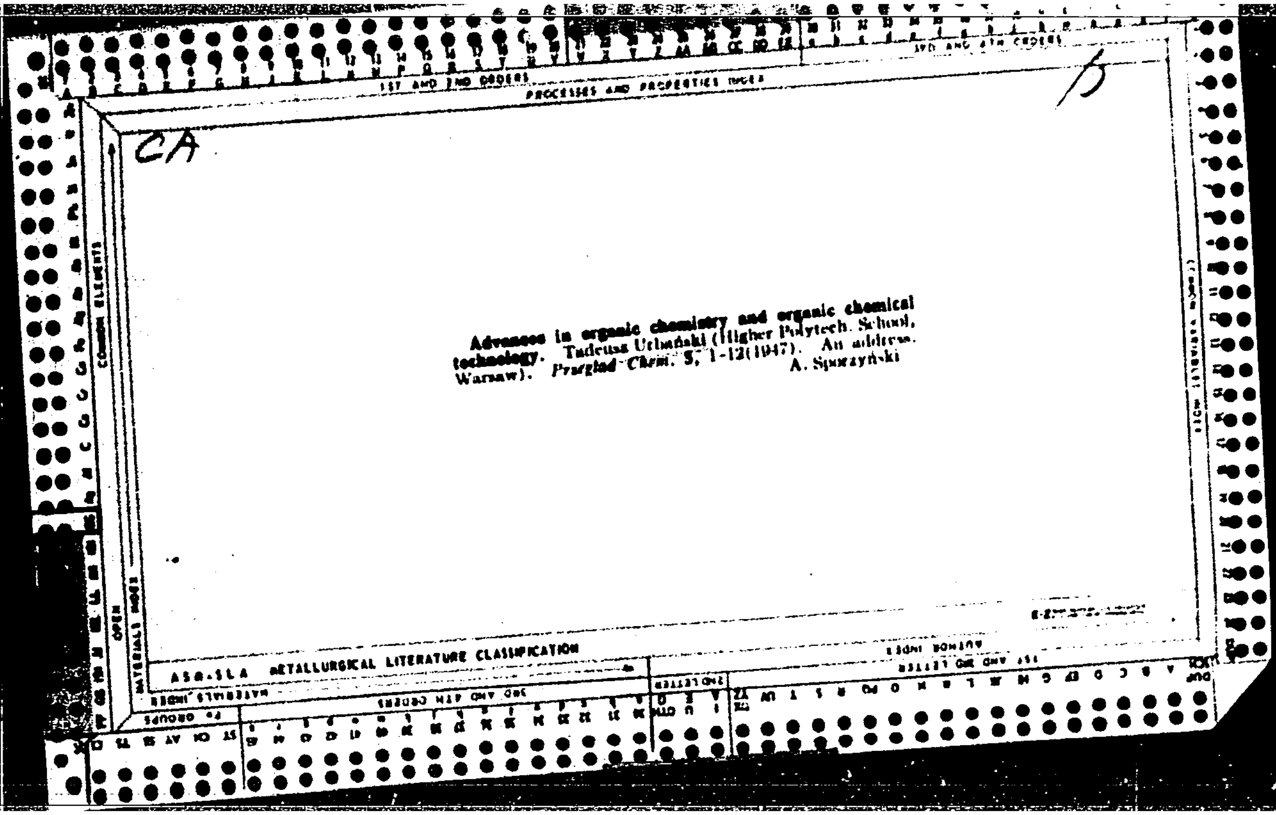
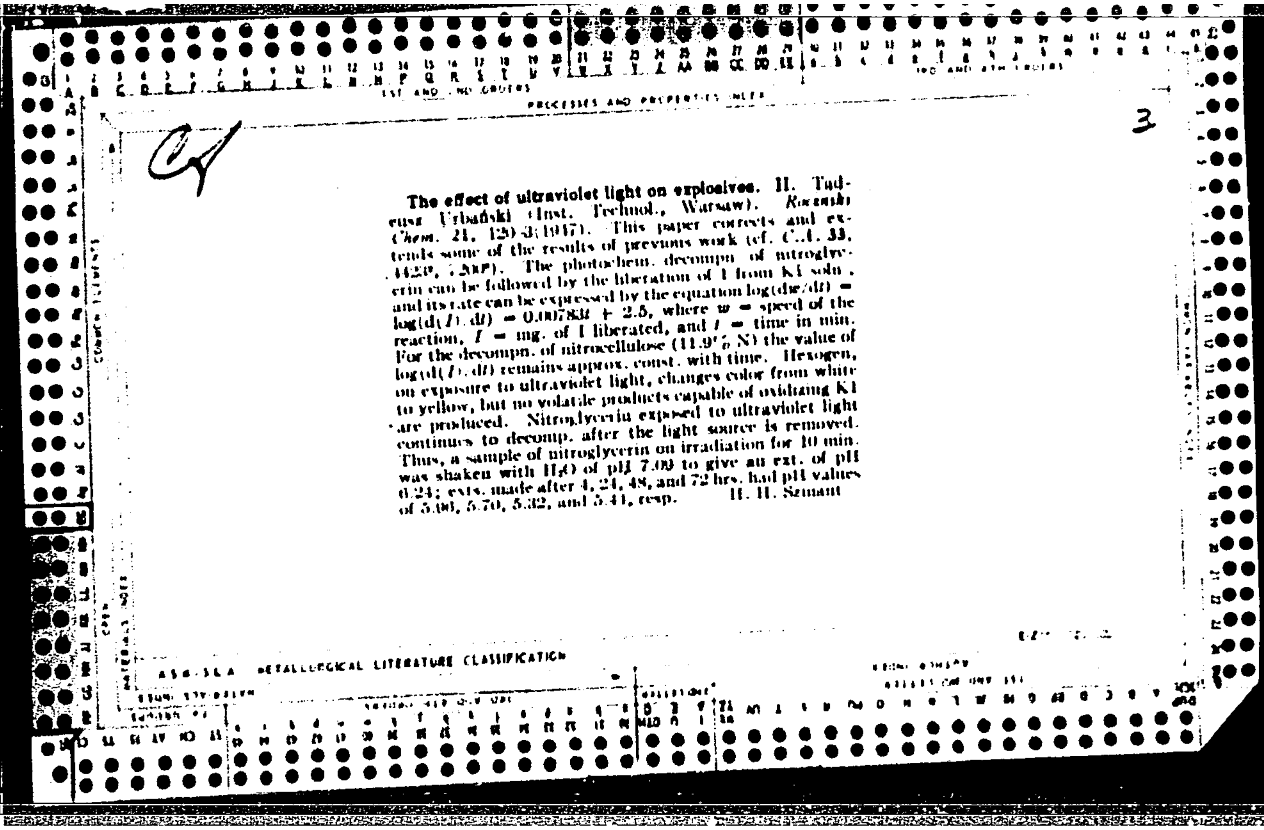


CA

15

Advances in organic chemistry and organic chemical technology. Tadeusz Urbański (Higher Polytech. School, Warsaw). *Przeglad Chem.* 5, 1-12(1947). An address.

A. Sporzyński



1ST AND 2ND GROUPS

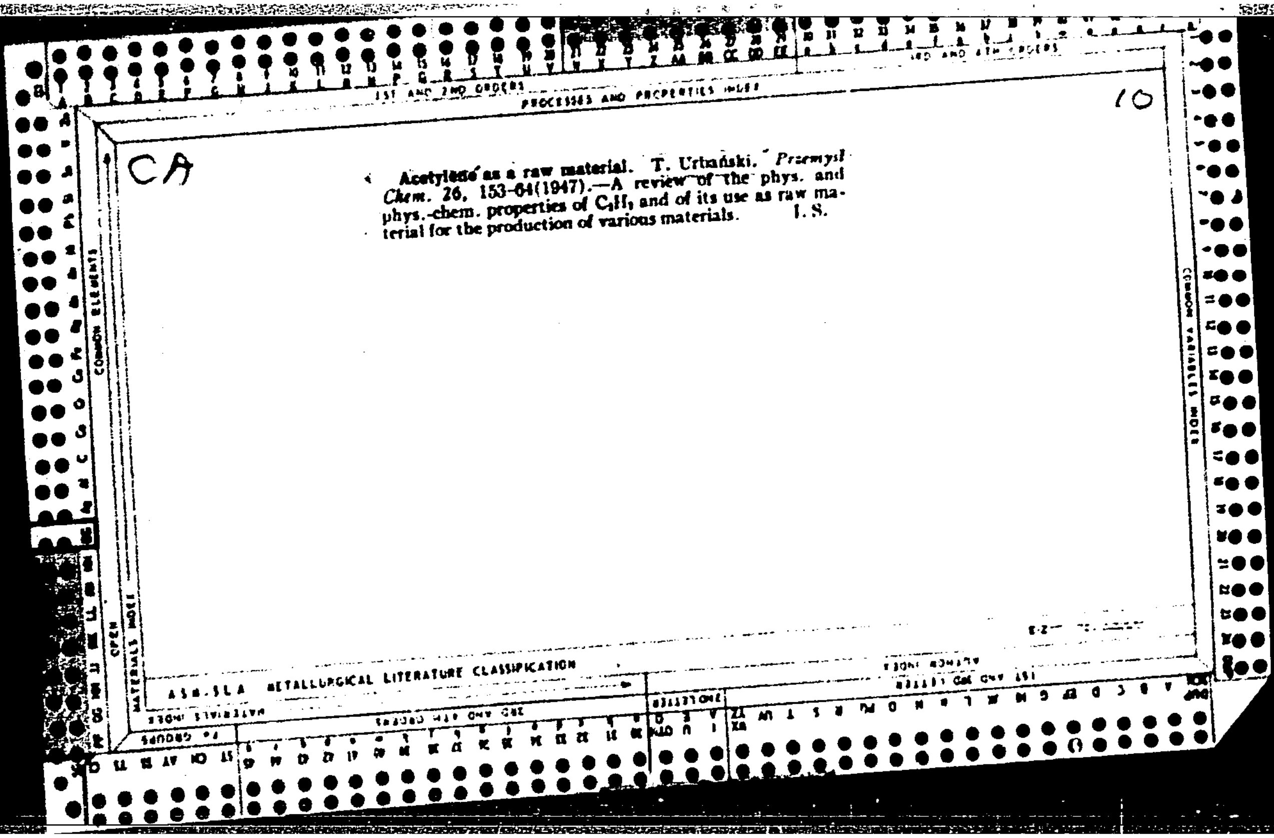
PROCESSES AND PROPERTIES INDEX

Ca

3

The effect of ultraviolet light on explosives. II. Tadeusz Urbański (Inst. Technol., Warsaw). *Roczniki Chem.* 21, 120-3(1947). This paper corrects and extends some of the results of previous work (cf. C.A. 33, 1420, 7558). The photochem. decompn. of nitroglycerin can be followed by the liberation of I from KI soln., and its rate can be expressed by the equation log(dw/dt) = log(d(I)/dt) = 0.0078t + 2.5, where w = speed of the reaction, I = mg. of I liberated, and t = time in min. For the decompn. of nitrocellulose (11.9% N) the value of log(d(I)/dt) remains approx. const. with time. Hexogen, on exposure to ultraviolet light, changes color from white to yellow, but no volatile products capable of oxidizing KI are produced. Nitroglycerin exposed to ultraviolet light continues to decomp. after the light source is removed. Thus, a sample of nitroglycerin on irradiation for 10 min. was shaken with H_2O of pH 7.00 to give an ext. of pH 6.24; exts. made after 4, 24, 48, and 72 hrs. had pH values of 5.00, 5.70, 5.32, and 5.41, resp. H. H. Szmant

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION



10

CA

Acetylene as a raw material. T. Urbański. *Przemysl Chem.* 26, 153-64(1947).—A review of the phys. and phys.-chem. properties of C_2H_2 and of its use as raw material for the production of various materials. I. S.

1ST AND 2ND ORDERS

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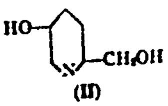
3RD AND 4TH ORDERS

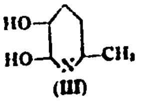
CA

118

Chemistry of pyridoxine (vitamin B_6). J. Urbański. *Przemysł Chem.* 26, 193-200(1947).—Early work on Vitamin B_6 is summarized. The present state of knowledge on the chemistry of pyridoxine is described. Japanese workers have heated hexoses with NH_4 salts in an autoclave at 150-160°. From the products of the reaction were derived the following 3 formulas:


(I)


(II)


(III)

These 3 formulas are derivs. of 3-pyridol. II contains a CH_2OH group characteristic of pyridoxine. This work indicates the reaction of sugars and NH_4 salts to compds. similar to vitamin B_6 and suggests a similar reaction in living organisms. U. advances a suggestion relative to the biosynthesis of pyridoxine from formaldehyde and N compds. This suggestion is based on the work of Japanese workers on the formation of 3-pyridol derivs. indicated above and upon U's work on the condensation of 3-pyridol with formaldehyde to 6-(hydroxymethyl)-3-pyridol. The production of ψ-pyridoxine or pyridoxamine in a living organism is discussed. Since pyridoxine plays a role in Fe metabolism, its use in the treatment of anemia is also discussed.

Jeannette Skarbek

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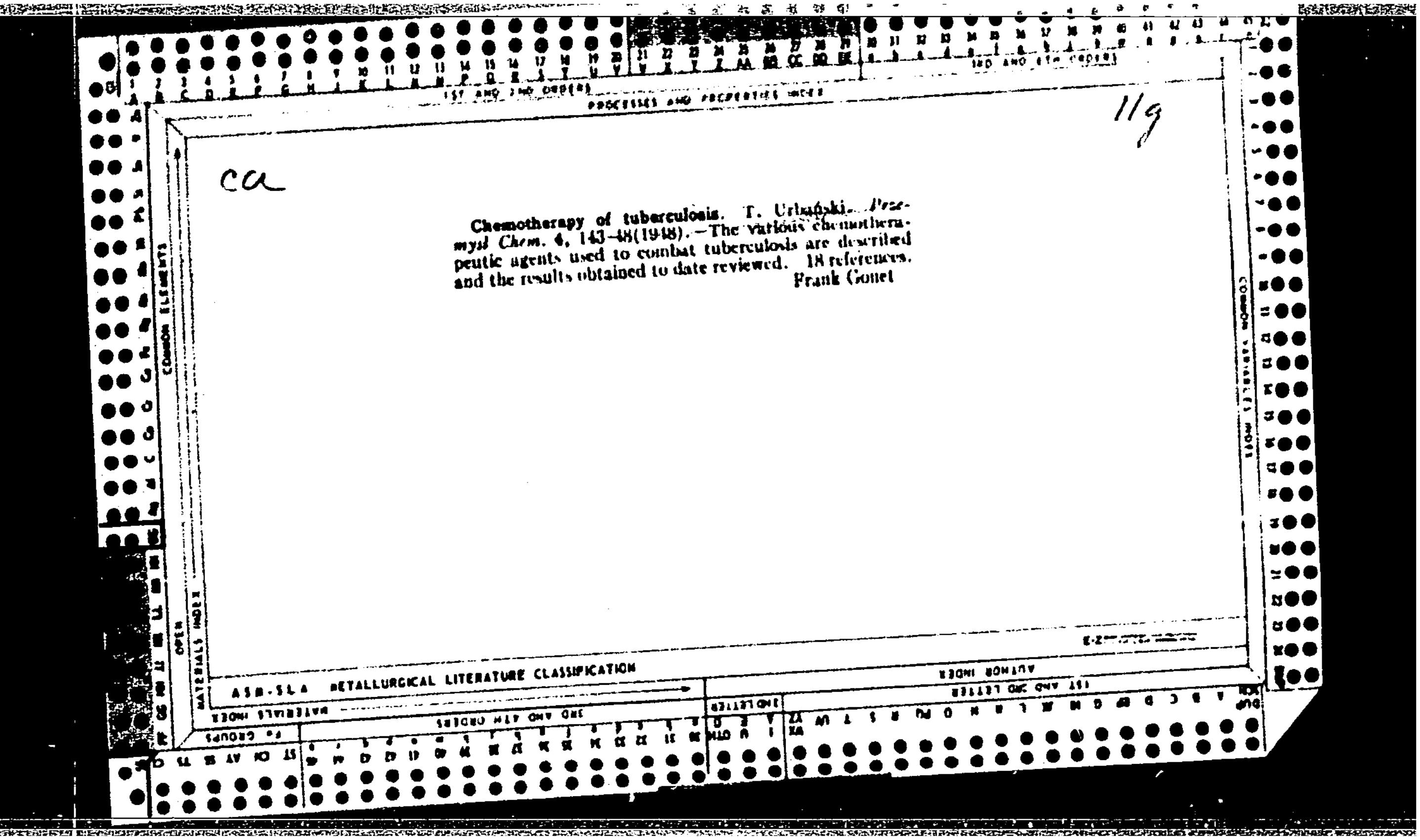
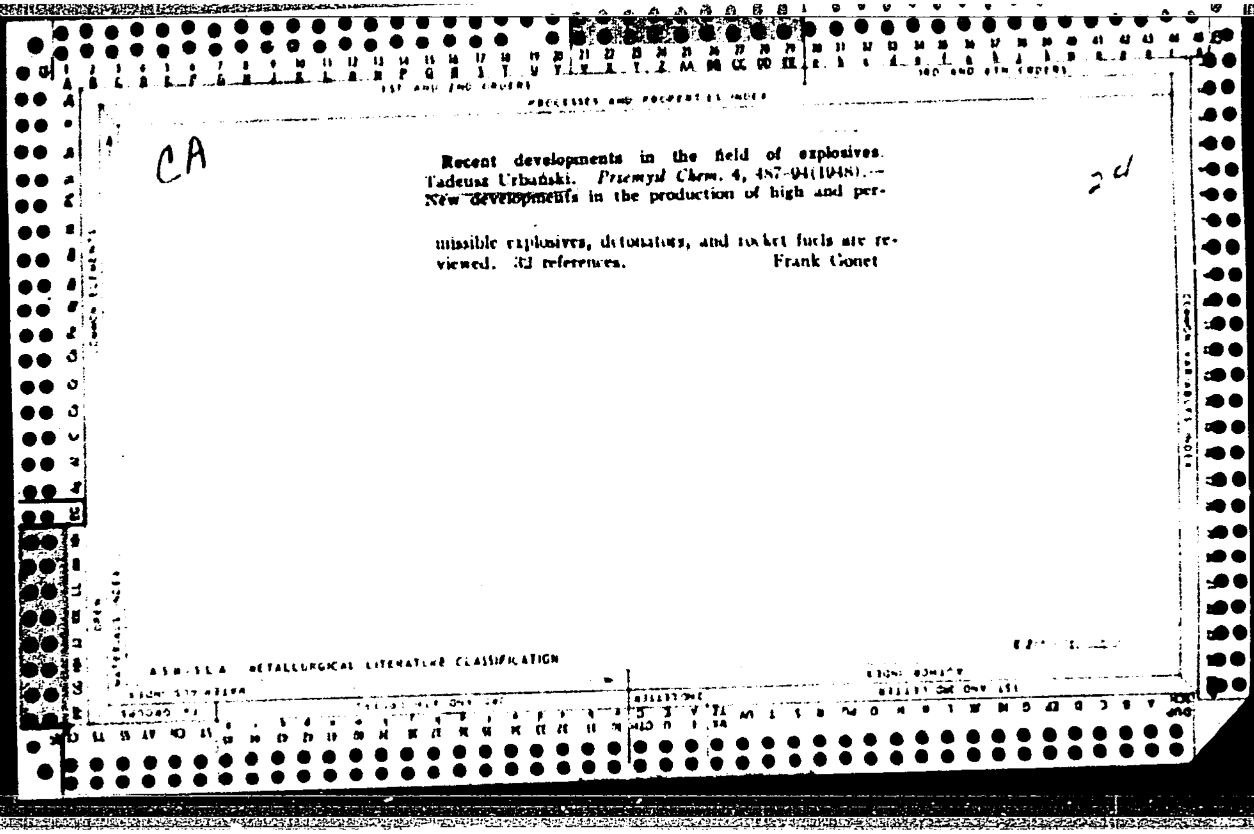


11g

ca

Chemotherapy of tuberculosis. T. Urbański. *Przemysl Chem.* 4, 143-48(1948).—The various chemotherapeutic agents used to combat tuberculosis are described and the results obtained to date reviewed. 18 references.

Frank Gonet



CA

2d

Recent developments in the field of explosives. Tadeusz Urbański. *Przemysl Chem.* 4, 487-94(1948).—New developments in the production of high and permissible explosives, detonators, and rocket fuels are reviewed. 33 references. Frank Gonet

CA

Chemotherapy of tuberculosis. II. T. Urbański. *Przemysl Chem.* 5(28), 457-66(1949); cf. *C.A.* 42, 7801c.—Recent advances in the chemotherapy of tuberculosis are reviewed and the structure and phys. properties of a large no. of agents are described. 39 references. Frank Gonet

CA

/ H

Salicylhydroxamic acid as an antitubercular agent. T. Urbański (Inst. Technol., Warsaw, Poland). *Nature* 166, 267-8(1950).—Salicylhydroxamic acid, (prepd. according to Jeanrenaud, *Ber.* 22, 1272(1889)) m. 168° (slow heating), 176-8° (quick heating), was converted to its monosodium salt by treatment with $NaHCO_3$. The Na salt was sol. in H_2O and a 0.1 *N* soln. gave pH 7.7. The bacteriostatic concn. of the Na salt was 1.0 mg. per 100 ml., the same as sodium *p*-aminosalicylate, *in vitro* in Youmans medium against *Mycobacterium tuberculosis* ($H_{37}Rv$ strain). The compds. were tested *in vivo* by using white mice inoculated (intravenously) with 0.1 mg. of *M. tuberculosis*. The compd., no. of animals, daily dose (mg. per mouse), mortality, av. tuberculous index, and av. survival time (days), resp., were: sodium *p*-aminosalicylate, 50, 10, 50, 58, and 23.1; sodium salicylhydroxamate, 50, 5, 62, 68, and 22.1; control, 50, —, 100, 100, and 20.1. The min. lethal dose of sodium salicylhydroxamate on white mice was about 12 times the curative dose.

Wesley H. Hartung

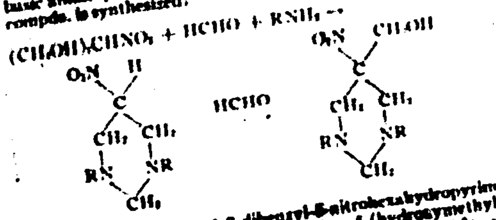
URBANSKI, T.

Salicylhydroxamic acid as a possible antituberculous agent. Gruzlica, Warszawa 18 no.2:206-208 Apr-June 1950. (CLML 20:7)

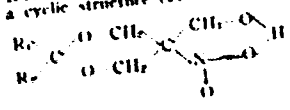
1. National Institute of Tuberculosis, Lodz.

CA

Reactions of nitroparaffins. IV. Reactions of nitromethane with formaldehyde and amines. Stanisław Malinowski and Tadeusz Urbański (Politech., Warsaw, Poland). *Roczniki Chem.* 25, 183-212(1951); cf. *C.A.* 42, 1766; 44, 4866i.—The reactions between $MeNO_2$, HCHO, and NH_3, even in dil. soln. and 0° lead to polymers of basic character. Replacing $MeNO_2$ by its di- or trihydroxymethyl derivative, or NH_3 by $MeNH_2$ gives the same results. However, from a substituted nitromethane and less basic amines, a series of hexahydropyrimidine and m-dioxane compds. is synthesized:



When R is $PhCH_2$, 1,3-dibenzyl-5-nitrohexahydropyrimidine, m. 57°, and 1,3-dibenzyl-5-nitro-5-(hydroxymethyl)hexahydropyrimidine, m. 124° (HCl salt, m. 180°; dioxalate, m. 162°), are formed in an exothermic reaction. When R is iso-Pr, 1,3-diisopropyl-5-nitro-5-(hydroxymethyl)hexahydropyrimidine, m. 140° (HCl salt, m. 145°; dioxalate, m. 139°), is isolated, and when R is cyclohexyl, 1,3-dicyclohexyl-5-nitro-5-(hydroxymethyl)hexahydropyrimidine, m. 104°, is formed. The monoesters of tris(hydroxymethyl)nitromethane (I) react slower than the above compds., requiring heating to 60°, and their hydrolysis during the reaction leads to the same products as from the bis(hydroxymethyl) derivs. (II). I are synthesized by conversion of $(CH_2OH)_3CNO_2$ (III) to acetals or ketals; these are esterified and hydrolyzed (dil. HCl/EtOH). III reacts with BzH (steam-bath, trace of concd. H_2SO_4) to give 2-phenyl-5-(hydroxymethyl)-5-nitro-m-dioxane, m. 125-6°, and is esterified to the acetate, m. 91°, and benzoate, m. 116-17°. III with anhyd. Me_2CO in the presence of anhyd. $CuSO_4$, at room temp. produces 2,2-dimethyl-5-(hydroxymethyl)-5-nitro-m-dioxane, m. 133-4°; acetate, m. 64°; or benzoate, m. 117-18°. $PhCH_2CHO$ and III (steam bath, trace of concd. H_2SO_4) form (2-phenylethyl)-5-hydroxymethyl-5-nitro-m-dioxane, m. 137-8°. Acetals and ketals are resistant to alk. hydrolysis (boiling with 15% KOH or NaOH) while acid hydrolysis (5% HCl at 60°) is complete in 5 min.; they are neither methylated by Me_2SO_4, nor oxidized by alk. $KMnO_4$ or H_2O_2; on heating with MeONa no HCHO is released. Also no acetals are formed from II. Therefore a cyclic structure (IV) is suggested. The al



(IV)

group in the 5-position can be aminated, giving 2-substituted-5-aminomethyl-*m*-dioxane compds. The following series is synthesized. *2-Phenyl-5-nitro-m-dioxanes*: 5-aminomethyl, m. 121-2° (HCl salt m. 152-3°); 5-dimethylaminomethyl-1,3-dioxane, m. 81-5°; 5-methylaminomethyl, m. 85-6° (HCl salt, m. 184-6°; nitroso deriv., m. 140°). *2,2-Dimethyl-5-nitro-m-dioxanes*: 5-aminomethyl, m. 160-1° (HCl salt, m. 172°); 5-methylaminomethyl, m. 153° (nitroso deriv., m. 111-12°); 5-dimethylaminomethyl, m. 87-8°; 5-anilinomethyl, m. 130-1° (nitroso deriv., m. 123-4°); 5-(*p*-carboxyanilinomethyl), m. 207°, (decompn.) (does not form a nitroso deriv.). Also 2-phenyl-5-(*p*-carboxyanilinomethyl)-5-nitro-*m*-dioxane, m. 208° (HCl salt (V), m. 222°; *N*-Ac deriv., m. 193°). Heating of the HCl salts breaks the dioxane ring to form (aminomethyl)bis(hydroxymethyl)nitromethanes and to liberate an aldehyde; 3-(*p*-carboxyanilino)-2-nitro-1,2-propanediol, m. 175-6° (HCl salt, m. 181-2°), is obtained from V. Details of the syntheses and soly. data of all compds. are given.

V. Reactions of primary and secondary nitroparaffins with ketene and with acetic anhydride. Tadeusz Urbański and Wanda Gurzyńska. *Ibid.* 213-21.—PrNO$_2$ (I) in the presence of anhyd. NaOAc with ketene (II) at 100-10° gives a volatile blue product (III) and MeCH(N:O)OAc or Ac(EtCO)NOAc, formed by direct acetylation. The purified product (after distn. of unreacted I, ether extn., neutralization of the ext., H$_2$O washes, and distn.) is a colorless oil, b[illegible] 125° b[illegible] 118°, n_D^{20} 1.4354 (yield, 12% based on I). Me$_2$CHNO$_2$ (IV) in the presence of anhyd. NaOAc with II at 70-80° gives III and O-acetyl-*aci*-2-nitropropane, Me$_2$C:N(→O)OAc (V) which, purified as above, b. 131°, b$_{12}$ 81-2°, n_D^{20} 1.4185, and gives HOAc on hydrolysis with 25% NaOH. V is also formed from IV with Ac$_2$O. I. Z. R.

RBANSKI, T.

8

Derivat[illegible] of qu[illegible]line. Preparation and properties of 5-[illegible] sulfohydroxylamino-8-hydroxyquin[illegible] Tadeu[illegible] Urbański (Inst. Technol., Wa[illegible] [illegible] (1951).—In an abstr. of this paper (C.A. 44, 4565) NaHSO$_3$ should be replaced by Na$_2$S$_2$O$_4$ in every case.

Also lines 35-40 inclusive should read as follows: "(4) [illegible] of 11.5 g. [illegible] adding 50 g. Na$_2$S[illegible] II obtained [illegible] II [illegible] + III [illegible] VI [illegible] III dissolved [illegible] and [illegible] (NH_4)[illegible]

URBANSKI, Tadeusz

reject

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

③ Chem

Penta- and hexanitrates of sorbitol. Tadeusz Urbański and Stanisława Kwiatkowska (Inst. Technol., Warsaw). *Roczniki Chem.* 25, 312–14(1951)(English summary).—The nitration of sorbitol gives a mixt. of penta- and hexanitrates; however, a nitration temp. of −10° favors the formation of the penta deriv., while the hexa deriv. is formed at 3–5°. To 15 g. sorbitol in 75 g. HNO_3 (d. 1.52), 150 g. H_2SO_4 (or 20% oleum) was added dropwise at 0–3° for 1.5 hrs.; from the cryst. 29.7 g. product, m. 38–45°, was obtained 8 g. sorbitol hexanitrate, m. 54.5–55°, by fractional crystn. from 50% aq. alc.; from the fraction easily sol. in 50% aq. alc., was pptd. 2 g. pentanitrate by adding H_2O to make an approx. 35% alc. soln. Sorbitol (5 g.) added dropwise to 50 g. concd. H_2SO_4 at −10 to −70° gave 7.5 g. of a mixt., mostly pentanitrate. The hexanitrate is easily denitrated by warming it in alc. pyridine. Gene A. Wozny

11 N

CA

Antitubercular activity of some 8-hydroxyquinoline derivatives. T. Urbánski, S. Slopek, and J. Venulet (Inst. Technol., Warsaw, Poland). *Nature* 168, 29(1951).—T28 was prepd. by action of $NaHSO_3$ on 5-nitroso-8-hydroxyquinoline to yield the monohydrate of *N*-sulfo-*N*-[5-quinolyl-8-hydroxy]hydroxylamine. *In vitro* expts. on rat heart showed T28 has no effect on the heart when 0.1 ml. of 5% soln. is administered. Intravenous doses of 50 mg./kg. body wt. to rabbit showed little effect. Guinea pigs inoculated with *Mycobacterium tuberculosis* and subsequently treated with T28 showed a tubercular index of 64, as compared with streptomycin 57. Other derivs. tested were 5-sulfo-8-hydroxyquinoline, 8-hydroxyquinoline sulfate, 5-amino- and 5,7-diaminoquinoline. The latter two were prepd. by reduction with $NaHSO_3$ of 5-nitroso- and 5,7-dinitro-8-hydroxyquinoline. T. J. Winnick

1952

URBANSKI, T.

Polish Technical Abst.
No. 1 1954
Chemistry and Chemical Technology

2630 663.11 : [illegible]

Urbański T., Chechelska B. Preparation on Laboratory Scale of Ergosterol from Mycelium Aspergillus Niger.

„Otrzymywanie ergosterolu z grzybni Aspergillus Niger w skali laboratoryjnej". (Prace Gł. Inst. Przem. Roln. i Spoż. No. 3) Warszawa, 1952. PWT. 4 pp., 1 tab.

The content of ergosterol in Aspergillus Niger taken from a citric acid factory was determined. Two principal methods of extraction of ergosterol were used: 1) extraction of fats and sterols and the hydrolysis of fats, followed by the isolation of ergosterol from the nonhydrolized portion, 2) hydrolysis of the whole material with potassium hydroxide in hydrated alcohol and the extraction of the unhydrolized portion with a suitable solvent. The evaporation of the solvent left the residue of ergosterol. In order to find the most convenient parameters. a number of experiments, in modification of both methods were carried out: the quantity of potassium hydroxide, the time of hydrolysis and extraction, the kind and the quality of solvent. The most suitable method was found to be: hydrolising the fungus with KOH in hydrated alcohol, using 30% of KOH — calculated on the basis of the dry fungus — mixing with water, filtering the precipitate and extracting the solution three times with benzene. Then the fungus was extracted three times with alcohol and the solution was filtered hot. The precipitate from benzene and alcoholic extract was recrystallized several times from the mixture of alcohol and benzene (4 : 1) or dichloroethane. Different kinds of fungi yielded various quantities of ergosterol. It is possible that the yield depends on the conditions of fermentation (medium, aeration) and of the age of fungi. The content of the crude ergosterol was 0.12 — 0.17%. After purification, it decreased to 0.08—0.12%. As the tests were executed on laboratory scale i.e. with small quantities of fungus, the losses were relatively great.

URBANSKI, TADEUSZ

4

1/2

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

Phenolic reactions of 3-hydroxypyridine. Halina Bojarska-Dahlig and Tadeusz Urbański. *~~Prace Placówek Nauk.-Badawcz. Ministerstwa Przemysłu~~ Chem.* 1952, No. 1, 1-15 (3) (English summary).—The authors undertook a systematic study of *3-hydroxypyridine* (I), the prototype of vitamin B_6. The NH_4 salt of I was obtained in 75.4% yield by adding dropwise 3 moles pyridine (b. 114-16°) to 255 cc. 65% oleum in presence of 6.6 g. Hg; the reactor, equipped with air condenser, was cooled with water, and then slowly brought to 280°, and kept at this temp. for 8 hrs. The reaction mixt. dissolved in water, neutralized, and concd. gave 308 g. NH_4 salt of I, crystals from water, m. 238-42°. The NH_4 salt (1 mole) fused with 9 moles KOH for 3 hrs. at 180° gave, after neutralization and extn. with Me_2CO, 80% of I, m. 124.5-5.0°. I was characterized by its salts: *hydrochloride*, b_{14} 204-5°, b_{25} 207.5-9.0°; *picrate*, m. 200-1.5° (from alc.); *picrolonate*, $C_5H_5ON.C_{10}H_8O_5N_4$, m. 241.5-3.0° (from alc.); *$C_5H_5ON.HgCl_2$*, m. 162.0-2.5° (from water); *$C_5H_5ON.Cu(OAc)_2$*, m. 180-3° (from water). Carboxylation of I gave hydroxypicolinic acids. Dry Na salt of I and CO_2 heated rapidly to 280° at atm. pressure gave 8-8.6% *3-hydroxypicolinic acid* (II). With slow heating less or no II was obtained. At 215-20° and 45 atm. the yield of II was 22%. Same conditions with K salt of I gave 24% *5-hydroxypicolinic acid* (III) and 3% of II. I mixed with anhyd. K_2CO_3 treated with CO_2 at 215° and 45 atm. for 8-9 hrs. gave III in yields which depended on the ratio of K_2CO_3 to 3-hydroxypyridine: ratio 1.5 gave highest yield, 85-7%; ratio 1.0 gave 70%. *$HgCl_2$ salt of* II, crystals from water, m. above 220° (decompn.). II *picrate* m. 158.5-62.0° (from water). *Me 3-hydroxypicolinate*, m. 73-4°. *Et 3-hydroxypicolinate* (IV), b_{15} 124°, b_{11-5} 152-4°, b_{17} 162°; *$HgCl_2$ salt*, m. 147-7.5° (from water), *picrate*, m. 118-19.5° (from water). *$HgCl_2$ salt of* III, m. 254-4° (from water). III *picrate* m. 205-6.5° (from water). *Me 5-hydroxypicolinate* m. 71.5-3.0° (from $CHCl_3$); *$HgCl_2$ salt*, m. 193.5-4.5° (from water). Esters and ethers of I were prepd. and characterized. I and Ac_2O gave 95% *3-pyridyl acetate*, b_{14} 92°, b_{45} 137°; *picrate*, m. 155.5-7.0° (from alc.); *picrolonate*, m. 166.5-7.5° (from alc.), *$HgCl_2$ salt*, m. 148-9° (from water). I and BzCl gave 81% *3-pyridyl benzoate*, m. 50-50.5° (from alc.); *picrate*, m. 152-3° (from $CHCl_3$); *$HgCl_2$*

(over)

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salt, m. 168-70°. I and Me_2SO_4 gave 92% *3-pyridyl Me ether*, b. 178-9°; *$HgCl_2$ salt*, m. 110-11°. K salt of I and PhBr in presence of I and $CuCO_3$ gave *3-pyridyl Ph ether* (V). Best yields (55-60%) of V were obtained with I-PhBr molar ratio of 1:1.5, 25% excess of I over its K salt, 2.5 g. $CuCO_3$ per mole of K salt, and heating reaction mixt. for 3 hrs. at 150° and 15 hrs. at 180°. V *hydrochloride* m. 95-7°; *picrate*, m. 130-2° (from alc.), *picrolonate*, m. 102-4° (from alc.); *$HgCl_2$ salt*, m. 76-7.5° (from alc.). I coupled with p-$O_2NC_6H_4N_2Cl$ and the azo compd. reduced with $SnCl_2$ gave 54.5% *2-amino-3-hydroxypyridine*, m. 163-7.5° (from C_6H_6-alc.); *picrate*, m. 257°. Iodination of I in mild alk. soln. gave 90% *2-iodo-3-hydroxypyridine*, m. 195-6° (from MeOH). I failed to undergo nitrosation, reaction with CH_2ClCO_2H to give (3-pyridoxy)acetic acid, or the Reimer-Tiemann reaction. $C_5H_4N.Hg(NO_3)_2$ did not give hydroxynitropyridine with HNO_3. Janina R. Spencer

URBANSKI, T; SERAFINOWA, B; MALINOWSKI, S; SLOPEK, S; KAMIENSKA, I; VENULET, J; JAKIMOWSKA, K.

Research on new drugs for the treatment of tuberculosis. Gruzlica, Warsz. 20 no.2:157-170; contd. Mar-Apr 1952. (CLML 22:3)

1. Of the Department of Chemotherapy of the Institute of Tuberculosis (Director--Prof. J. Misiewicz, M. D.).

URBANSKI, T.; SERAFINOWA, B.; MALINOWSKI, S.; SLOPEK, S. KAMIENSKA I.; VENULET, J.;
JAKIMOWSKA, K.

Research on new drugs in the treatment of tuberculosis; thiosemicarbazones. Gruzlica, Warsz. 20 no.3:292-302; concl. May-June 1952.
(CIML 23:2)

1. Of the Chemical Laboratory of the Institute of Tuberculosis (Director --Prof. J. Misiewicz, M.D.), Warsaw.

URBANSKI, T.; MALINOWSKI, S.

Synthesis of antituberculous drugs at the Institute of Tuberculosis.
Gruzlica 20:6 Suppl. 2:81-91 1952. (CLML 24:2)

1. Of the Laboratory of the Syntheses of Therapeutics of the Institute of Tuberculosis, Warsaw.

Urbański, Tadeusz

Chemistry of pyridine. III. On the carboxylation of 3-hydroxypyridine with carbon dioxide. Halina Bojarska-Dahlig and Tadeusz Urbański (Inst. Technol., Warsaw). *Roczniki Chem.* 26, 158-67(1952) (English summary); cf. C.A. 41, 5129*d*; 49, 1033*c*.—NH_4 3-pyridinesulfonate (1 mole) was fused with KOH (9 moles) at 180° for 3 hrs., the melt dissolved in water, adjusted to a pH of about 10 with concd. HCl and then to about 4.5 with HOAc, and the resulting ppt. extd. with Me_2CO to give 80% 3-hydroxypyridine (I), m. 124.5-5.0° (from water); *HCl salt*, b_{11} 201-5° and b_{20} 207-9°; *picrate*, m. 200.0-1.5°; *picrolonate*, m. 241.5-3.0°; *$HgCl_2$ complex*, m. 162°; *$Cu(OAc)_2$ complex*, m. 190-9° (decompn.). After I as the Na salt (II) (4.75 g.) was heated to 220°, dry CO_2 (at atm. pressure) was introduced while the temp. was raised to 280° during 30 min., kept there 6-9 hrs., the mixt. dissolved in 25 ml. water, acidified with concd. HCl, filtered, neutralized with $NaHCO_3$, the unreacted I filtered off, the filtrate acidified with HOAc, and 3-hydroxypicolinic acid (III) pptd. as the *Cu salt* (IV) from the boiling filtrate on satn. with NaOAc. IV was decompd. with H_2S and two purifications were effected through IV to yield 0.56-0.60 g. III, m. 203-4°; *picrate*, m. 159-62°; *$HgCl_2$ complex*, m. 220° (decompn.). III (2.6 g.) as the Ag salt was refluxed 8.5 hrs. with 1.25 g. EtI in 24 ml. dry C_6H_6, the AgI filtered off, and the benzene soln. evapd. to yield 2.39 g. *Et 3-hydroxypicolinate*, b_{21} 162° and b_3 121°; *picrate*, m. 118-19°; *$HgCl_2$ complex*, m. 147°. Dry CO_2 was introduced to 1.9 g. I, as the Na salt, in a 50-ml. autoclave to a pressure of 40-45 atm., the whole heated during 1 hr. to 210-20°, kept at this temp. for 9 hrs., and the resulting mixt. worked up as above yielded 0.62 g. III. I (1.9 g.) as the K salt was treated with CO_2 under pressure, the mixt. heated as above 8 hrs., the product dissolved in 20 ml. water, the soln. sepd. from carbonized substances by filtration, neutralized with concd. HCl, and acidified with HOAc to give crystals of *5-hydroxypicolinic acid* (V). The filtrate was neutralized with $NaHCO_3$, any sepd. I filtered off, and after acidifying with HOAc, the Cu salts of the β-hydroxypicolinic acids were pptd. with $Cu(OAc)_2$ from hot soln. The Cu salts were decompd. with H_2S and two fractions with different water solubilities were obtained. The less sol. fraction consisted of V (0.75 g.); the total yield of V was 24%, m. 267-8°; *picrate*, m. 265.0-6.5°; *$HgCl_2$ complex*, m. 253-4°; *Me ester*, m. 72-3°; *$HgCl_2$ complex of Me ester*, m. 194°. The more sol. fraction was III (3%). I (0.02 mole) was mixed with K_2CO_3 (0.03 mole) and the mixt. treated in an autoclave with dry CO_2 (40 atm.) at 215° for 8.5 hrs. From the reaction product, worked up in the usual way, was obtained 2.73 g. V. When I and K_2CO_3 were mixed in molar amts. and treated as above 70% V and 1% III were obtained. Larger excesses of K_2CO_3 did not improve the yield of V.

T. T. Galkowski

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OPALICKI, TADEUSZ

Urbanski, Tadeusz

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√ **Reactions of nitroparaffins. VII. Reactions of nitromethane with acetaldehyde and amines.** Zygmunt Eckstein and Tadeusz Urbański (Inst. Technol., Warsaw, Poland). *Roczniki Chem.* 26, 161-62(1952)(English summary); cf. C.A. 46, 7993e.—Addn. of AcH to $MeNO_2$ gave either $MeCH(OH)CH_2NO_2$ (I) or $O_2NCH(CHMeOH)_2$ (II), depending on the reaction conditions. AcH (88 g.) in 80 ml. water added with cooling to 15° [illegible] $MeNO_2$ and 0.1 g. K_2CO_3 (more K_2CO_3 was added during the reaction to keep the pH at 7.5-8.0) and the mixt. heated 3-6 hrs. at 50° gave 75-80% I, extd. with 1 l. Et_2O and purified through the Na salt, [illegible] oil, b. 70-4°/0.2-0.3 mm., n_D 1.4412. Chlorination of the Na salt of I gave 72-4% crude $MeCH(OH)CHClNO_2$ (III), b_3 89-91°. The Na salt (187 g.) of I in 880 ml. CCl_4 and 76.5 ml. Br in 200 ml. CCl_4 kept below 0° gave 76.5-81.5% crude $MeCH(OH)CHBrNO_2$ (IV), n_D 1.5[illegible]. I and IV cause skin burns and severe eczema. AcH (308 g.) in 236 ml. water added to 244 g. $MeNO_2$ and 1 g. $Ca(OH)_2$, with cooling to 30-5°, and the mixt. stirred 2-3 hrs., then left at room temp. 48 hrs. in a tightly closed vessel (pH = 6.5-7.0 at end of the reaction), treated with CO_2 and 6-7 l. Et_2O, and the dried Et_2O layer distd. at 0.1 mm. Hg pressure gave 80-95% (based on $MeNO_2$) crude II, not crystd., n_D 1.4695. AcCl (25 g.) and 15 g. II in 50 ml. $CHCl_3$ refluxed until evolution of HCl ceased gave, after distn., a residue of semicryst. product which, filtered, yielded 8.6 g. $O_2NCH(CHMeOAc)_2$, needles, m. 87-8° (from alc.). III and AcH gave 61.3% $O_2NCCl(CHMeOH)_2$ (V), m. 118-20°. Freshly distd. AcH (18 ml.) in 18 ml. water and 36.8 g. IV neutralized (litmus) with aq. KOH, the mixt. let stand overnight, salted out with NaCl, extd. with Et_2O, and the product crystd. from CCl_4 and $CHCl_3$ (1:1) gave white needles of $O_2NCBr(CHMeOH)_2$, m. 111-13°. Cyclic acetals (*m-dioxanes*) of the type $\underbrace{O.CHMe.CR^2(NO_2).CHMe.O.CR^1R^2}$ (VA) were prepd. from II by 2 methods: (1) 1 mole crude II and 1.5 moles aldehyde was added [illegible] concd. H_2SO_4, the mixt. heated several hrs. at [illegible] and the product isolated by addn. of Et_2O or alc.; (2) 1 mole crude II or V and 1.5 moles aldehyde in C_6H_6 were added 0.05-0.1 g. $PhSO_3H$, alc. HCl, and H_2SO_4, the water was [illegible] throughout the course of the reaction, and the [illegible] isolated as in (1). The following *2-substituted-5-nitro-4,6-dimethyl-m-dioxanes* (VA, $R^1 = R^2 = H$; R^2, m.p., and % yield given) were prepd. by method (1): *Me* (VI), [illegible], 71.4; *CCl₃* (VII), 101-6°, 25.7; *Ph* (VIII), 126-7°, 17.5 [37.2% by method (2)]; *p-O₂NC₆H₄* (IX), 115-7°, 41.0;

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*p-HO*C$_6$H$_4$(X), 222-4°, 15.7; *p-MeOC$_6$H$_4$* (XI), 132-[illegible]°, 14.9; *PhCH$_2$CH$_2$* (XII), 94.5-6°, 3.7; *p - Me$_2$NC$_6$H$_4$* (XIII), 181-3°, 8.9. In the prepn. of XI-XIII unreacted aldehyde was removed with aq. NaHSO$_3$ before isolation of the product. The VA were crystd. from alc. (VII, IX, XII, XIII), from aq. (1:1, 1:3) alc. (VI, XI), or from a mixt. (3:2) of C$_6$H$_6$ and alc. (X). They are resistant to dil. alkali (except VII), but hydrolyze in dil. acids. They give pseudonitroles with HNO$_2$ while II with HNO$_2$ splits off AcH to give the nitrolic acid. The following VA (R^1 = R^2 = *H;* R^3, m.p., and % yield given) were prepd. by method (2): *p-ClC$_6$H$_4$*, 107-9°, 30.4; *o-HOC$_6$H$_4$* (XIV), 144-6°, 21.2; *p-AcNHC$_6$H$_4$*, 206-7.5°, 13.2. *m-Dioxanes* (VA, R^1 = *H;* R^2, R^3, m.p., and % yield given): *p-O$_2$NC$_6$H$_4$, Br*, 142-4°, 32.5; *p-MeOC$_6$H$_4$, Br*, 124-6°, 9.2; *Ph, Br*, 98-100°, 36.5; *p-O$_2$NC$_6$H$_4$, Cl*, 125-7°, 55.6; *Ph, Cl*, 91-2.5°, 36.5. All the VA derived from 4,6-dimethyl-*m*-dioxane are white cryst. compds. except XIII, which is yellow. XIV required 16 hrs. for its prepn. and could be made by method (2) only. VA [R^1 = R^2 = Me, R^3 = H (XV), and R^1 = Me, R^2 = Pr, R^3 = H (XVI)] were prepd. Crude II (150 g.), 600 ml. dry Me$_2$CO, 200 g. anhyd. CuSO$_4$, and 1 ml. alc. HCl (30%) refluxed 24 hrs., the sulfate filtered off, and the Me$_2$CO distd. gave an oily residue which, washed with water and distd. up to 79°/5 mm., yielded a cryst. residue of 40 g. (21.1%) XV, needles with camphoric odor, m. 73.5-5.5° (from alc.). II (30 g.), 80 g. PrAc, 100 g. anhyd. CuSO$_4$, and 1 drop alc. HCl 16 hrs. at 100° gave 15 g. (28%) XVI, needles with camphoric odor m. 68-70° (from 1:1 aq. alc.). Attempts to synthesize tetrahydroöxazine or hexahydropyrimidine derivs. from II and primary amines were unsuccessful since II decompd., splitting off AcH and forming resinous polymers. Cyclohexylamine and III gave AcH and a cryst. unstable product, m. 62-4°, presumably the salt of cyclohexylamine and the hydroxamic acid [ClC(O)NHOH].

[illegible] R. Spencer

URBANSKI, T.

Reactions of aromatic amines with cyanoguanidine. Formation of aryl derivatives of amidinourea and their transformation into carbanilides. T. Urbański, B. Skowrońska-Serafinowa, H. Dąbrowska, and T. Kubicka (Inst. Technol., Warsaw). *Bull. acad. polon. sci.*, Classe III, 1, 71-4 (1953)(in English).—p-$O_2NC_6H_4NH_2$ (I) boiled with $NCNHC(:NH)NH_2$ (II) in 12% HCl yields p-$O_2NC_6H_4NHC(:NH)NHC(:NH)NH_2$ (III). When I and II are boiled with 22% HCl, p-$O_2NC_6H_4NHCONHC(:NH)NH_2$ (IV), m. 231-2°, is formed. III was converted to IV by boiling in 22% HCl. p-$RC_6H_4NHCONHC(:NH)NH_2$, when R = H (V), m. 166-177°; R = OH (VI), m.p. of HCl salt, about 250°; R = COOH (VII), m. 198-200°. Boiling with aniline cleaves IV, V, and VI to the corresponding p-$RC_6H_4NHCONHC_6H_5$ and $NH_2C(:NH)NH_2.HCl$. VII is converted to *carbanilide*. IV shows strong bacteriostatic action against staphylococci in vitro. Charlotte S. Russell

URBANSKI, T.

"On the Structure of Some Aliphatic Nitro Compounds." In English. P. 239,
(GEODEZJA I KARTOGRAFIA, Vol. 1, No. 6, 1953, Warszawa, Poland.)
(Polska Akademia Nauk.)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 3,
No. 12, Dec. 1954, Uncl.

URBANSKI, T.

URBANSKI, T.

Hydroxamic Acids. III. Analogs of salicylohydroxamic acid. Tadeusz Urbański, Stanisław Malinowski, [illegible] Henryk Piotrowski [illegible] Roczniki [illegible] [illegible] 26, 553 [illegible] following new hydroxamic acids all possess low [illegible] and bacteriostatic action on mycobacteria, particularly compds. no. T106 and T139. *Salicylohydroxamic acids*: 3-*Me* (T95), m. 149-50°; 4-*Me* (T97), m. 190-2°; 5-*Me* [illegible] 185°; [illegible] T112, m. 154-6°; [illegible] T143 [illegible] 44°. *1-Hydroxy-2-naphthohydroxamic acid* [illegible] *hydroxynaphthohydroxamic acid* [illegible] 2°. Products T95, T97, T100, T112, and T143 were synthesized according to Jean Renaud (Ber. 22, 1270(1889)). Yields for the salicylo derivs. ranged from 35 to 60%. Yields averaged 25% for T106 and T139. Products T161, T106, and T139 were synthesized from NH_2OH in alc. KOH with the Me esters of the corresponding CO_2H acids.

Clayton F. Holoway

URBANSKI, T

[illegible]

HCl [illegible]

$C_{?}H_{6}O_{2}N_{?}$·HCl m.p. 282–284 [illegible]

[illegible]

247–248 [illegible]

URBANSKI, T.

Preparation of [illegible]

Zbigniew [illegible]

[illegible]

existing methods of synthesis [illegible] 4-ethylpyridine (II) (C.A. 42, ...ide (I), viz., in the prepn. of [illegible] III Et ester [illegible] isonicotinic acid (III) (C.A. 43, 3826), [illegible] (IV) (C.A. 39, [illegible]), and the hydrazide (Meyer and Mally [illegible] The main improvements are [illegible] of the [illegible] followed by fra[illegible] made [illegible] solution of II [illegible]

[illegible] washing of the pptd. I with alc. and [illegible] although the 1st crop can be applied directly to clinical use.

Clayton F. Holoway

URBAŃSKI, TADEUSZ

Reactions of cyanoguanidine with aromatic amines. III. 2,4-Dihydroxy-7-nitroquinazoline from 4-nitroanthranilic acid and cyanoguanidine. Tadeusz Urbański, Barbara Skowrońska-Serafinowa and Jadwiga [illegible] (Inst. Technol. Tuberculosis, Warsaw). *Roczniki Chem.* 27, 167-9 (1953)(English summary); cf. *C.A.* 48, 13017*d*. When heated with HCl, 4-nitroanthranilic acid and cyanoguanidine yield 2,4-dihydroxy-7-nitroquinazoline (I), tautomeric with 2,4-dioxo-7-nitrotetrahydroquinazoline. I has been prepd. in another way by Huntress and Gladding (*C.A.* 37, 650^9).

Clayton F. Holoway

URBANSKI, TADEUSZ

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✓Investigation of hydroxamic acids. II. 5-Bromosalicylhydroxamic acid. Tadeusz Urbański and Wiesław Lewenstein (Polytech., Warsaw). *Roczniki Chem.* 27, 314(1953); cf. *C.A.* 49, 2308h.—Br (10 g.) in 50 ml. of glacial HOAc was added slowly to 6.3 g. of salicylhydroxamic acid in 100 ml. of glacial HOAc at 60°. Excess Br was removed with $NaHSO_3$ and, after cooling, the product was distd., washed with cold water, and dried to yield 35 g. crude 5-bromosalicylhydroxamic acid, white crystals, (from alc.), m. 232° (decompn.), poorly sol. in water and solvents such as ether, acetone, CCl_4, benzene, and xylene at boiling temps., readily sol. in boiling alc. and HOAc; it has a bitter taste. C. F. Holoway

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[illegible], T.

Some derivatives of 4,4'-dihydroxydiphenyl sulfone. Kazimierz Okoń and Tadeusz Urbański. *Roczniki Chem.* 27, 348-65(1953)(English summary).—*o*-Cresol (37.1 g.) is added over 30 min. to 9.8 g. H_2SO_4 at 35–40°, the mixt. heated slowly to 100° for 8 hrs., the pressure reduced from 350 to 100 mm. while the temp. is raised to 180° over 30 hrs. The H_2O formed in the reaction is removed by distn. The temp. is then lowered to 110°, 2*N* NaOH added to pH 9, filtered, and the filtrate adjusted to pH 3.5 with HCl, resulting in 23.05 g. of a mixt. of *4,4'-dihydroxy-3,3'-dimethyldiphenyl sulfone* (I) and its *2',4-dihydroxy isomer* (II), m. 262-8°. The mixt. is dissolved in boiling Me_2CO, twice its vol. of warm C_6H_6 is added, and the mixt. is cooled, pptg. 72% I, m. 271-1.5°; concn. of filtrate yields 24% II, m. 220-1°. Similarly, pyrocatechol at 70–80° with a digestion temp. of 210° for 24 hrs. gives 28% of *3,3',4,4'-tetrahydroxydiphenyl sulfone*, m. 237–40°. 4,4'-Dihydroxydiphenyl sulfone (III) (50 g.) is added to 250 ml. H_2SO_4 at 30°, cooled to 15°, and 250 ml. HNO_3 is added, not permitting the temp. to rise above 35–40°. The mixt. is heated at 60–70° for 30 min., dild. with an equal vol. of H_2O, and filtered, giving the *3,3'-dinitro analog* of I, m. 231–3° (from EtOH), 31 g. of which is dissolved in 820 ml. of 25% aq. NH_3 at 60°, 127 g. $Na_2S_2O_4$ is added, the mixt. heated on a H_2O bath for 6 hrs. and cooled, to give 23.8 g. of the *3,3'-diamino analog*, m. 270–1°. *4,4'-Dihydroxy-3-carboxydiphenyl sulfone*, m. 299-300°, is obtained in 12% yield by treating salicylic acid with H_2SO_4 (as in the prepn. of I) at 200°. Heating 25 g. III and 250 ml. Ac_2O with 0.1 g. NaOAc on a H_2O bath for 8 hrs. gives the *di-Ac deriv.* of I, m. 170-1.5°. III (25 g.) is dissolved in 100 ml. 2*N* NaOH and 20 g. Me_2SO_4 is added slowly, pptg. 92% *4,4'-dimethoxydiphenyl sulfone*, m. 132-3° (from EtOH). III (25 g.) is dissolved in a soln. of 17 g. NaOH in 75 ml. H_2O at 50–60°, 19 g. $ClCH_2CO_2H$ is added, and the mixt. heated at 100° for 1 hr. The resulting ppt. is dissolved in boiling H_2O, and cooled, pptg. *4,4'-di(carboxymethoxy)diphenyl sulfone* (V), m. [illegible] Heating 3.66 g. V with 200 ml. MeOH-[illegible] of the [illegible] *ester*, m. 132-3°. [illegible]

URBANSKI, T

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droxyquinone and [illegible]

[illegible]

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[illegible]emistry of pyridine. IV. Reactions of amides of [illegible]nic, nicotinic, and isonicotinic acids with formaldehyde and amines. Bożena Chechelska and Tadeusz [illegible] (Inst. Technol., Warsaw). Roczniki Chem. 27, [illegible] (English summary).—[Throughout this abstr. [illegible] nicotinoyl, R' = picolinoyl, and R'' = isonicotinoyl.] [illegible] acid amide (24.4 g.) in 80 ml. H_2O, 2.2 g. K_2CO_3, [illegible] g. 37% CH_2O are heated on a H_2O bath for 1 hr., [illegible]CH_2OH (I), m. 141-2°. I *picrate*, m. 140-2°, [illegible] I.*HCl*, m. above 120° (decompn.), are also prepd. [illegible]CH_2[illegible] (II), m. 244-8°, is made by slowly adding 2.4 [illegible] acid amide to 11.5 ml. 85% H_2SO_4 and 1.9 g. 37% [illegible] warmed at 40° for 1 hr., and holding at [illegible] 20-30°; 1.03 g. product ppts. after treat[illegible] with Na_2CO_3. Similarly 1.5 g. I treated with H_2SO_4 [illegible] 1.03 g. of II. Nicotinic acid nitrile (3.5 g.) [illegible] 2.1 g. 37% CH_2O and 25 ml. 85% H_2SO_4 [illegible] heated at 30° for 2 hrs., poured into ice [illegible] and Na_2CO_3 added to pH 5, the pptd. starting material is filtered off, and addnl. Na_2CO_3 ppts. 1.3 g. II. Also prepd.: II *picrate*, m. 224-6°, and II.*HCl*, m. 287-9°. In the same manner, *R'NHCH₂OH* (III), m. 164-6° (*HCl* salt, m. 202-4°) and *(R'NH)₂CH₂* (IV), m. 124-6°, are obtained. *R'NHCH₂NMe₂.HCl* (V), m. 159-61°, is prepd. (80%) by refluxing 2.4 g. $R'NH_2$ with 2.1 g. Me_2NH·HCl and 2.5 g. 37% CH_2O in 15 ml. anhyd. EtOH for 3 hrs.; V *picrate* m. 180-2°. III (1.7 g.) and 1.7 g. β-naphthol are refluxed in 20 ml. EtOH, contg. 1 ml. conc. HCl, for 1/4 hr., the EtOH is distd., and the residue allowed to stand for several months, giving the *2-C₁₀H₆OCH₂NHR'*, m. 170-7.5°. By the methods described, *R''NHCH₂OH* (VI), m. 326-8°, VI *picrate*, m. 145-7°, *(R''NH)₂CH₂* (VII), m. 253-7°, *R''NHCH₂*-138-8° (decompn.), VII *picrate*, m. 166-8°, and VIII *picrate*, m. 183-6°, *NMe₂.HCl* (VIII), are prepd. V and VIII show a slight bacteriostatic action in vitro against *Mycobacterium smegmatis* and *Mycobacterium* 379. [illegible] Mark

Urbanski Tadeusz

4.5 g. [illegible] EtOH [illegible] N-dimethylaminoethyl [illegible] obtained by [illegible]

URBANSKI, T.

CH

The structure of some aliphatic nitro compounds. T. Urbański, *Bull. acad. polon. sci.*, Cl. III, 2, 593-4(1954)(in Engl.); cf. *C.A.* 49, 8002a.—Certain aliphatic nitro compds. do not show the 270 mμ absorption characteristic of the nitro group. An explanation involving H bonding is suggested (cf. Albrecht and Corey, *C.A.* 33, 4845⁹; Shugam, *C.A.* 45, 4112b). Two examples cited are $[Me_2C(NO_2)CH_2]_2NH_2^+Cl^-$ and $[MeC(CH_2OH)(NO_2)CH_2]_2NH$. J. E. A.

MU

URBANSKI, T.

3652 547.551.43 : 547.495.9

Urbański T., Skowrońska-Serafinowa B., Dąbrowska H. **Reactions of Cyanguanidine with Aromatic Amines. Preparation of New Derivatives of Amidine-Phenyl-Urea, their Transformation into Diphenyl Urea Derivatives.** CH

„Reakcje cyjanoguanidiny z aminami aromatycznymi. Otrzymywanie nowych pochodnych amidyno-fenylo-mocznika i przekształcenie ich w pochodne dwufenylomocznika". Roczniki Chemii (PAN). No. 3, 1954, pp. 423—437.

Continuing the work on the reactions of aromatic amines with cyanguanidine in the presence of hydrochloric acid, a number of new derivatives of amidine-phenyl-urea were obtained, corresponding to the general formula: $X.C_6H_4NH\text{-}CO\text{-}NH\text{-}C(NH)\text{-}NH_2$. It was found that boiling the amidine-urea derivatives in aniline led to the splitting of these compounds and the formation of various diphenyl-urea derivatives. A hypothesis is advanced for a chain reaction mechanism.

②

Urbański, T.

Reactions of aromatic amines with cyanoguanidine. Formation of derivatives of amidinourea and their reaction with aniline. T. Urbański, B. Skowrońska-Serafinowa, and H. Dabrowski. *Bull. acad. polon. sci.*, Classe III, 2, 455-9 (1954); cf. *C.A.* 49, 8807.—The following 3,4-$XYC_6H_3NHCONHC(:NH)NH_2$ were prepd. (X, Y, and m.p. given): Cl, H, 143-4° (I); Br, H, 172-3° (II); NH_2, H, (HCl salt, m. 300°) (III); SO_3H, H, 267-9° (IV); SO_2NH_2, H, 312-13° (V); H, OH, — (sulfate, m. 230-2°) (VI). The compds. were made by boiling the corresponding amines with cyanoguanidine in the presence of HCl. V was formed from sulfanilamide-HCl in aq. medium without added HCl. The reaction with aniline is exemplified by: $p\text{-}XC_6H_4NHCONHC(:NH)NH_2 + PhNH_2 \rightarrow p\text{-}XC_6H_4NHCONHPh$ (VII) $+ (H_2N)_2C{:}NH$; VII $+ PhNH_2 \rightarrow (PhNH)_2CO + p\text{-}XC_6H_4NH_2$. I-VI were tested against *Mycobacteria* for bacteriostatic action with inhibiting concns. varying from 1-125 mg.-%, depending upon the deriv. used and the strain of organism. Howard Nechamkin

URBANSKI, Tadeusz

Production of alginic acid from Fucus vesiculosus. Bożenna Chodkiewicz and Tadeusz Urbański. *Prace Głównego Inst. Przemysłu ~~Rolnego i Spożywczego~~* 4, No. 2, 37-42 (1954).—*F. vesiculosus* gathered in July and August has an alginic acid content of 12% dry basis. Of a no. of extn. and purification methods investigated, the following was found the most satisfactory. Seaweed is washed with 0.5% HCl, extd. 4 times with 2% Na_2CO_3, the ext. is bleached with ClO_2 or $NaClO_2$, alginic acid is pptd. with HCl and washed with water and alcohol. — Alina S. Szczesniak

URBANSKI, Tadeusz; MALINOWSKI, Stanislaw; SKOWRONSKA-SERAFINOWA, Barbara;
CHECHELSKA, Bozenna; DABROWSKA, Halina; FALECKI, Jerzy; GURNE,
Daniela; HALSKI, Leszek; SLOPEK, Stefan; KAMIENSKA, Irena;
VENULET, Jan; JAKIMOWSKA, Krystyna; URBANSKA, Alicja

Search for new antituberculous agents. Gruzlica 22 no.10:681-690
Oct 54.

1. Z Oddzialu Syntezy Lekow Instytutu Gruzlicy; kierownik prof. dr.
T.Urbanski, dyrektor: prof. dr. J.Misiewicz.
(CHEMOTHERAPY, in various diseases
tuberc., progr.)
(TUBERCULOSIS, therapy
antituberc. agents, research)

URBANSKI, T.

POL

Reactions of aliphatic nitro compounds. X. Formation of the hexahydropyrimidine ring using 1-nitropropane, formaldehyde, and ammonia. Tadeusz Urbański, Zbigniew Bieruncki, and Ewa Lipska. *Roczniki Chem.* 28, 169-74 (1954)(English summary); cf. preceding abstr. $PrNO_2$, CH_2O, and NH_3 in a 1:3:3 molar ratio at 65-6° for 1.5 hrs. give 3% *5-nitro-5-ethylhexahydropyrimidine* (I), m. 159-60°. A 12.5% yield of I is obtained by dissolving 55 g. 2-nitro-2-ethyl-1,3-propanediol in 120 ml. 25% NH_3, extg. with C_6H_6 for several weeks and treating with alc. HCl to obtain I.HCl, m. 159°, which reacts with 30% $NaNO_2$ to give the *1,3-dinitroso* deriv., m. 116°. Boiling this with HCl gives the dihydrochloride of I, m. 157-8°. **XI. A new derivative of tetrahydroöxazine with nitromethane, formaldehyde, and benzylamine.** Tadeusz Urbański and Hans [illegible]. *Ibid.* 175-84.—One mole of $(HOCH_2)_3CNO_2$ and 1 mole of benzylamine with app. 3 moles of 30% CH_2O at 15° warmed for 3 hrs. at 65°; after the initial reaction subsides there is obtained 42% *5-nitro-5-hydroxymethyl-3-benzyltetrahydro-1,3-oxazine* (I), m. 142-4°. To 7.5 g. I and 4 g. NaOH in 20 ml. H_2O is slowly added 30 ml. 30% H_2O_2 at 20°, the mixt. stirred for 1 hr., decompd. with HOAc, extd. with Et_2O, and evapd. Treatment of the oily residue with alc. HCl gives 2 g. *5-nitro-3-benzyltetrahydro-1,3-oxazine.HCl*, m. 210-12°. I (4 g.) reduced with 3.5 moles H in the presence of Pd gives the *5-amino* deriv. Chester Placek

URBANSKI, T.; GURNE, A.

"Reactions of Aliphatic Nitro Compounds. XI. A New Derivative of Tetrahydroxazine with Nitromethane, Formaldehyde, and Benzylamine", P. 175, (ROCZNIKI CHEMII, Vol. 28, No. 2, 1954, Warsaw, Poland)

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

URBAN [illegible]

Ch

✓**Reactions of cyanoguanidine with aromatic amines. V. Preparation of new derivatives of 1-phenyl-3-amidinylurea, their transformation into diaryl urea derivatives.** Tadeusz Urbański, Barbara Skowrońska-Serafinowa, and Halina Dalkowska (Inst. Technol., Warsaw). *Roczniki Chem.* **28**, 423-37(1954)(English summary); cf. *C.A.* 49, 860*f*.—The following p-$RC_6H_4NHCONHC(NH_2):NH$ were obtained: R = Cl (I), m. 143-4°; R = SO_3H (II), m. 267-9°; R = NH_2 (III), m. > 300° (hydrochloride). Boiling of I with $PhNH_2$ gives p-$ClC_6H_4NHCONHC_6H_5$, but on prolonged boiling I yields p,p'-dichlorocarbanilide. The corresponding unsym. carbanilides from II and III could not be obtained. I shows a strong bacteriostatic action against saprophytic mycobacteria. R. Dowbenko

R

② MGT

URBANŚKI, T.

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POL.

New thiosemicarbazones. T. Urbański and Cz. Belżecki
(Inst. Technol. Warsaw). Roczniki Chem. 28, 677-8
(1954)(English summary).—Thiosemicarbazones of the
followi g acids were prepd.: *p*-acetamidobenzoylformic, m.
190°; *p*-hydroxybenzoylpropionic, m. 102°; *p*-acetamido-
benzoylpyruvic, m. 178-80°; and of the *Et esters* of the
following substituted acetic acids: -nitrobenzoyl, m.
108°; *p*-aminobenzoyl, m. 145°; nicotinoyl, m. 217°;
isonicotinoyl, m. 102°; and *p*-acetamidobenzoylpyruvic
acid, m. 125°. No preparative details given. The compds.
are being tested for tuberculostatic activity. C. P.

Urbański, T.: Teoria nitrowania. Warszawa Państwowe Wyd. Naukowe. 1955. 130 pp. zł. 18.50.

Chem

Chem

Urbanski, T. The theory of nitration.

URBANSKI T.

3650 547.232 : 547.722.5 : 541.572 : 545.623

Urbański T., Ciecierska D. On Aliphatic Nitrocompounds. The Structure of some Aliphatic and Heterocyclic Nitrocompounds on the Basis of Ultraviolet Absorption Spectrum. CH

„O nitrozwiązkach alifatycznych. Budowa niektórych nitrozwiązków alifatycznych i heterocyklicznych na podstawie badań widma absorbcji w ultrafiolecie". Roczniki Chemii (PAN). No. 1, 1955, pp. 11—21, 5 figs., 1 tab.

The authors examined the ultraviolet adsorption spectra of 20 aliphatic and heterocyclic nitrocompounds, nitroparaffins and their derivatives, finding that certain nitrocompounds do not show the maximum of absorption in the proximity of λ = 370 mμ, which is characteristic for the nitro-group. The authors explain this by the formation of chelate rings through hydrogen bonds between both oxygen atoms of the nitro-group and the hydrogen atoms of at least two hydroxyl groups, or by the formation of hydrogen bonds between one of the oxygen atoms of the nitro-group and the hydrogen of the amino-group.

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URBANSKI, T.

Synthesis and degradation of some derivatives of tetrahydro-1,3-oxazine. D. Gurne and T. Urbański (Polish Acad. Sci., Warsaw). *Bull. acad. polon. sci.*, ~~Classe~~ III, 3, 175–8(1955)(in English); cf. *C.A.* 42, 1756.—RCH_2NO_2 CH with CH_2O and $PhCH_2NH_2$ gave the following *5-nitro-5-alkyl-3-benzyltetrahydro-1,3-oxazines* (I) (alkyl and m.p. given): *Me* (II), 66–8°; *Et* (III), 68–70°; *Pr* (IV), 46–8°. I(R = CH_2OH) (V), m. 140–2° (*C.A.* 49, 8320*h*), with NaOMe gave I (R = H) (VI), m. 44–6°. I heated 5 hrs. with concd. HCl under ultraviolet light or preferably with

1% HCl-EtOH gave the following *$HOCH_2C(NO_2)RNHCH_2$-Ph.HCl* (VII) (R and m.p. given): *Me*, 102–4° (*N,O*-di-Bz deriv., m. 112–14°); *Et*, 150–2° (*N,O*-di-Bz deriv., m. 105–7°); *Pr*, 136–8° (*N,O*-di-Bz deriv., m. 90–2°); *$HOCH_2$* 177° (decomps.) (*N,O,O-tri-Ac deriv.*, m. 98–100°); *H*, 150° (decomp.). VII warmed with CH_2O gave I. VII with MeONa gave the Na salts which with CO_2 gave the following *$O_2NCHRCH_2NHCH_2Ph$* (R, m.p. of HCl salt, and m.p. of *N*-tosyl deriv. given): *Me*, 148–50°, 82–4°; *Et*, 150–1°, 88–9°; *Pr*, 152–4°, 110–12°; *H*, 147° (decomp.), — (*N-Ac deriv.*, m. 193–5°). The structures were established by the analytical results, by formation of oily *N*-nitroso compds. which warmed with HCl gave the amines, and through the Bz, Ac, and tosyl derivs. J. E. A.

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URBANSKI, T

6

✓**Some properties of tetrahydro-1,3-oxazines derived from 1-nitrobutane or 1-nitroisobutane.** T. Urbański, J. Kolesińska, and H. Piotrowska (Polish ~~Acad. Sci.,~~ Warsaw). *Bull. acad. polon. sci.*, Classe III, 3, 179-82(1955)(in English); cf. *C.A.* 49, 13998*i*.—$PrNO_2$ (*C.A.* 42, 175*b*), $EtNO_2$ [*Roczniki Chemii* 26, 182(1952)], and $MeNO_2$ (*C.A.* 46, 7993*c*) react with CH_2O and NH_3 to form several ring compds. whose formation must be due to a reactive N—H group. But $BuNO_2$ or iso-$BuNO_2$ (1 mole) heated several hrs. on the steam bath with 3 moles CH_2O and 1 mole NH_3, dried and dissolved in alc.-HCl gave only *5-nitro-5-propyltetrahydro-1,3-oxazine-HCl* (I), m. 190-2° (*picrate*, m. 163-4°), or its *5-iso-Pr isomer-HCl* (II), m. 190° (*picrate*, m. 167-8°), and $HOCH_2C(NO_2)PrCH_2NH_2$ (III), m. 169-70° (HCl salt) (*O,N-dibenzoyl deriv.*, m. 101-2°), or its *iso-Pr isomer* (IV), m. 165-7° (HCl salt) (*picrate*, m. 158-60°). All attempts to combine further I and II with CH_2O failed, indicating low activity of the N—H group. I and II were also prepd. by heating 2-nitro-2-propyl-1,3-propanediol or the iso-Pr isomer with 1 mole CH_2O and 1 mole NH_3. I and II gave oily nitroso derivs. which, warmed with HCl gave I and II. The free bases I and II gave the corresponding *methiodides*, m. 199-200°, and 201-2°, resp. I and II boiled several hrs. with concd. HCl gave III and IV, resp. The free bases III and IV with CH_2O gave the bases I and II, resp. The bacteriostatic concn. of I against various *Mycobacteria* is 62.5-125 mg. % (cf. *C.A.* 47, 101*f*). Janet E. Austin

CH

URBANSKI, T.

Poland/ Organic Chemistry - Synthetic organic chemistry

E-2

Abs Jour : Referat Zhur - Khimiya, No 4, 1957, 11781

Author : Piotrowska H., Urbanski T.

Inst : Polish Academy of Sciences

Title : On Derivatives of 5-Nitrotetrahydro-1,3-Oxazine Substituted in Position 2.

Orig Pub : On the formation of derivatives of 5-nitrotetrahydro-1,3-oxazine substituted in position 2.
Bull. Acad. polon. sci., 1955, Cl.3.3, No 7, 389-390 (English)

Abstract : See RZhKhim, 1956, 39594.

Card 1/1

URBANSKI, T.

Anti-tubercular properties of some derivatives of 1 : 3-benzoxazine. T. Urbanski, D. Gürne, Z. Eckstein, and S. Slopek (*Bull. Acad. polon. Sci.*, III, 1955, 3, 397—399).—A number of benz-1 : 3-oxazine derivatives were prepared and shown to be bacteriostatic. The bacteriostatic concentrations of some of the compounds *in vitro* against saprophytic *Mycobacteria* are given, the 6-bromo-3-hexyl- and -3-benzyl-3 : 4-dihydro-deriv. being very effective although they have LD_{50} 3 g./kg. per os.

R. J. Magee.

CH

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URbANSKi, J.

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Reactions of 5-nitro-1,3-dioxane with diazo compounds and the synthesis of arylazonitro diols. Z. Eckstein and T. Urbański. *Bull. acad. polon. sci.*, Classe III, 3, 433-6 (1955); cf. Gochenour and Degering, *C.A.* 43, 4646*f*; U.S. 2,474,779 (*C.A.* 43, 8153*i*); U.S. 2,474,780 (*C.A.* 43, 8154*a*).—Derivs. of 5-nitro- (I) and 5-hydroxymethyl-*m*-dioxane (II) with diazo compds. formed azo compds. when the pH was maintained between 7.5-8.5 with KOH. II treated with base lost a mol. of CH_2O and the resulting azo compds. $R^1R^2C.O.CHR^3.C(NO_2)(N:NAr).CHR^3.O$ (III) were identical with those formed by I. The following III were obtained (R^1, R^2, R^3, Ar, m.p., % yield): *H, Ph, H, Ph*, 107-8.5°, 59.1; *H, Ph, H, p-ClC$_6$H$_4$*, *cis*-(IV), 111.5-113°, 21.6, and *trans*-IV, 165-7° (decompn.), 7.2; *H, Ph, H, p-O$_2$NC$_6$H$_4$*, 140-8° (decompn.), 27.8; *H, Ph, H*, *p*-MeC$_6$H$_4$, 125.5-27°, 58.2; *H, Ph, H, 2-C$_{10}$H$_7$*, *cis*-(V), 126.5-8.0°, 18.2, and *trans*-V, 182-3° (decompn.), 5.6; *H, Ph, Me, Ph*, 145-6°, 40.9; *H, Ph, Me, p-ClC$_6$H$_4$*, 155.5-6.5°, 66.4; *H*,

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Z. Eckstein...

Ph, Me, p-$O_2NC_6H_4$, 186-7° (decompn.), 52.4; H, Ph, Me, p-MeC_6H_4, 111-2°, 21.1; H, Ph, Me, 2-$C_{10}H_7$, 142-3° 31.9; H, p-ClC_6H_4, Me, p-$O_2NC_6H_4$, 187-8° (decompn.), 28.0; H, Ph, Pr, p-$O_2NC_6H_4$, 137-8.5°, 64.4; H, Ph, iso-Bu, p-$O_2NC_6H_4$, 127.5-8.5°, 18.5; Me, Me, Me, Ph, 99-100.5°, 20.5; Me, Me, Me, p-ClC_6H_4, 138.5-40°, 38.1; Me, Me, Me, p-$O_2NC_6H_4$, cis-(VI), 120-1°, 11.7, trans-VI, 163-4°, 29.6; Me, Me, Me, 2-$C_{10}H_7$, 112-14°, 21.0; Me, Me, H, Ph, 94-5°, 86.8; Me, Me, H, p-ClC_6H_4, 142-3.5° (decompn.), 91.8; Me, Me, H, p-$O_2NC_6H_4$, 173-4° (decompn.), 72.5; Me, Me, H, p-MeC_6H_4, 124.5-26°, 91.2; Me, Me, H, 2-$C_{10}H_7$, 133-4°, 86.4; Me, Me, p-$O_2NC_6H_4SC_6H_4$, 168-9° (decompn.), 73.5. The isomers of IV and V were sepd. by fractional crystn. and on hydrolysis gave the same arylazonitro diols. They also gave the same ultraviolet spectra. The following ultraviolet spectra data were obtained (max., E): cis-IV 291, 15,810, 405-6, 320; trans-IV 291-6, 12,260, 405, 261.4; cis-VI 286-8, 19,630, 430-5, 248.5; trans-VI 285, 18,010, 430-5, 233.5. Hydrolysis of III with alc. HCl gave the corresponding ketone or aldehyde, as well as $ArN{:}NC(CH_2OH)_2NO_2$ (VII). The following derivs. of VII were obtained (Ar, m.p., % yield): Ph, 97-9°, 82.0; p-ClC_6H_4, 97-8°, 72.0; p-$O_2NC_6H_4$, 114-16°, 73.0; p-MeC_6H_4, 95-7°, 65.0; and 2-$C_{10}H_7$, 107-8°, 68.9. VII warmed with BzH and the H_2O removed azeotropically yielded cyclic acetals. Francis Taylor, Jr.

2/2

FM

URBANSKI, T.

Eckstein, Z.; Urbanski, T. On the alkylation of derivatives of 5-nitro-1, 3=dioxane.
In English. p. 489.

MATEMATYKA

Vol. 3, No. 9, 1955 Warszawa, Poland

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 5, No. 10 Oct. 56

URBANSKI, T.

Urbanski, T. On the productions of the reaction of 1-nitropropane with formaldehyde and ethylendiamine. In English. p. 493.

MATEMATYKA

Vol 3, No. 9, 1955 Warszawa, Poland

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

URBANSKI, T.

Poland/Chemistry of High-Molecular Substances, F

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61691

Author: Urbanski, T.

Institution: None

Title: Conference on Macromolecular Chemistry at Zuerich

Original Periodical: Symposium z zakresu chemii makromolekularnej w zurychu, Wiadom chem., 1955, 9, No 12, 662-667; Polish

Abstract: None

Card 1/1

URBAŃSKI, T.

"Chemia i technologia materiałów wybuchowych" (Chemistry and technology of explosive materials), by T. Urbański. Reported in New Books (Nowe Ksiazki), No. 14, July 15, 1955

URBANSKI, T.

Skowronska-Serafinowa, B.; Dabrowski, H. Reactions of cyanoguanidine with aromatic amines. VI. Some new derivatives of amidine-phenyl-urea and their reactions with aniline. p. 450.
ROCZNIKI CHEMII, Warszawa, Vol. 29, no. 2/3, 1955.

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4, no. 10, Oct. 1955, Uncl.

URBANSKI, T

explain [illegible]
instead at 2 OH groups. U. and C. suppose that the cause is
more strong electron repelling character of the amino group.
A. Semenisov

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Reactions of aliphatic nitro compds. XIII. Liebermann reactions of secondary amines containing a nitro group. Tadeusz Urbański and Zygmunt Eckstein (Inst. Technol., Warsaw). *Roczniki Chem.* 29, 916-18(1955)(English summary); cf. *C.A.* 49, 11414*d*.—The Liebermann test was modified for secondary amines contg. nitro groups, which were found to interfere under the usual conditions. The soln. of nitrosamine in H_2SO_4 with phenol was not warmed. XIV. Action of nitroparaffins on reaction between 2-aminopyridine and formaldehyde. Tadeusz Urbański and Barbara Skowrońska-Serafinowa. *Ibid.* 367-74.—HCHO (I), 2-aminopyridine (II), and 1-nitropropane stirred together at 20-30° and allowed to stand overnight gave *5-nitro-5-ethyl-1,3-dioxane*, m. 52-4°, and *N,N'-di(2-pyridyl)methylenediamine* (III), m. 130-1° (methiodide, m. 225-6°), gives Liebermann test for secondary amine. III was also obtained by mixing nitro alcs. (2-ethyl-2-nitro-1,3-propanediol, 2-methyl-2-nitro-1,3-propanediol, and 2-methyl-2-nitropropanol) with II. The catalytic effect of primary and secondary nitroparaffins, $PhNO_2$, $CH_2(CO_2Et)_2$, HCl, PhOH, NH_4Cl, and β-hydroxypyridine on the reaction of I and II to give III was also studied. XV. Interpretation of ultraviolet absorption spectra of nitroparaffin derivatives. Tadeusz Urbański. *Ibid.* 375-8.—On the basis of ultraviolet spectra (near 270 mμ) of some aliphatic amino nitro compds. a suggestion based on analogy with amino acids was made that the H of an amino group can be bound with both O atoms of a nitro group by means of 2 H bonds. XVI. Products of reaction of 1-nitro-*n*-butane with formaldehyde and ammonia. Tadeusz Urbański and Hanna Piotrowski. *Ibid.* 379-91.—Mixing $BuNO_2$ (I), HCHO, and NH_3 in a molar proportion 1:3:1 gave after fractional crystn. from EtOH *5-nitro-5-propyltetrahydro-1,3-oxazine·HCl* (II), m. 190-2° (dimethiodide, m. 199-200°), and *2-nitro-2-(hydroxymethyl)pentylamine·HCl* (III), m. 169-70° (*O,N*-di-Bz deriv., m. 101-2°). Use of 2-nitro-2-propyl-1,3-propanediol (IV) instead of I gave higher yields of II and III. I treated with 3 moles HCHO in excess NH_3 at room temp. gave *5-nitro-5-propylhexahydropyrimidine·2HCl* (V), m. 171-3° (di-*N*-nitroso deriv., m. 99-100°). V warmed with aq. EtOH gave *2-nitro-2-propyl-1,3-propylenediamine*, m. 178-9° [di(*p*-nitrobenzoyl) deriv., m. 216-17°]. Using IV instead of I gave a better yield of V. IV warmed with excess NH_3 gave *3,7-dinitro-3,7-dipropyl-1,5-diazacyclooctane* (VI), m. 73-4° (mono-HCl salt, m. 173-4°; mono-*N*-nitroso deriv., m. 110-11°). VI.HCl was of moderate bacteriostatic activity *in vitro* against saprophytic mycobacteria. XVII. Products of reaction of 1-nitroisobutane with formaldehyde and ammonia. Tadeusz Urbański and Janina Kolesińska. *Ibid.* 392-8.—$Me_2CHCH_2NO_2$ (I) (1 mole) treated with 3 moles HCHO, and 1 mole NH_3 gave after fractional crystn. of their HCl salts from EtOH *5-nitro-5-isopropyltetrahydrooxazine* (II).*HCl*, m. 190° [yield

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10%; picrate, m. 167-8° (decompn.); N-Me MeI salt, m.
201-3°], and *2-nitro-2-(hydroxymethyl)isopentylamine* (III).-
HCl, m. 165-7° (yield 6%; picrate, m. 158-60°). II and
III were also prepd. by treating 1 mole *2-nitro-2-isopropyl-*
1,3-propanediol (m. 84-6°, obtained by heating I with
HCHO at pH 7.5) with 1 mole HCHO, and 1 mole NH_3.
II warmed on a steam bath with concd. HCl 6 hrs. gave
HCHO and III.HCl, while III warmed with HCHO gave
II. **XVIII. Products of reaction of nitromethane with**
isovaleraldehyde. Tadeusz Urbański, Zygmunt Eckstein,
and Wiesław Sabótka. *Ibid.* 399-409.—The residue from
the prepn. of 1-nitro-4-methyl-2-pentanol (I) according to
the method of Henry [*Rec. trav. chim.* 16, 201(1897)],
Mousset [*Rec. trav. chim.* 21, 95(1902)], or Bouveault and
Wahl [*Bull. soc. chim. France* 29, 643(1903)] was found to
contain *5-nitro-2,8-dimethyl-4,6-nonanediol* (II), m. 92-3°
(from CCl_4); diacetate, m. 108.5°. A yield of 6.5 g. II
was also obtained when 29.4 g. I was treated with 20 g.
isovaleraldehyde (III) in the presence of 1.6 ml. NEt_3.
Chlorination and bromination of the Na salt of I led to *1-*
chloro-1-nitro-4-methyl-2-pentanol (IV), b_1 98.5-9.0°, and *1-*
bromo-1-nitro-4-methyl-2-pentanol (V), $b_{1.2}$ 107-8°, resp.
Treatment of IV and V with excess III led to *5-chloro-5-*
nitro-2,8-dimethyl-4,6-nonanediol, m. 123-6 [illegible] CCl4),
and *6-bromo-5-nitro-2,8-dimethyl-4,6-nonanediol* (VI), m.
133-4° (from CCl_4-$CHCl_3$), resp. II reacted with alde-
hydes, yielding *2-R-substituted 5-nitro-4,6-diisobutyl-1,3-*
dioxanes; when R = *Ph*, *p-ClC₆H₄*, *p-MeOC₆H₄*, *p-O₂N-*
C₆H₄, the m.p. was 146, 160, 148, 176°, resp. Similarly,
VI reacted with benzaldehyde to give *2-phenyl-5-bromo-5-*
nitro-4,6-diisobutyl-1,3-dioxane, m. 153°. IV reacted with
HCHO in the presence of triethylamine to give *2-chloro-2-*
nitro-5-methyl-1,3-hexanediol, m. 103.5-4.0° (from CCl_4
and then C_6H_6). **XIX. Preparation of alkyd resins from**
nitrophthalic acids and ethylene glycol. Tadeusz Urbański
and Marceli Ficines. *Ibid.* 412-15.—4-Nitrophthalic acid
was esterified by $(HOCH_2)_2$ at 156-8°, yielding a resin more
readily than 3-nitrophthalic acid (I). Formation of a
seven-membered ring by means of a hydrogen bond between
the nitro group and the carboxyl group of I was suggested
as an explanation.

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So. East European Accessions List Vol. 5, No. 9 September 1956