

SHORT COMMUNICATIONS

A MODIFIED SYNTHESIS
OF FURAN-3,4-DICARBOXYLIC ACID

E. J. FORBES AND M. KHURSHID A. KHAN*

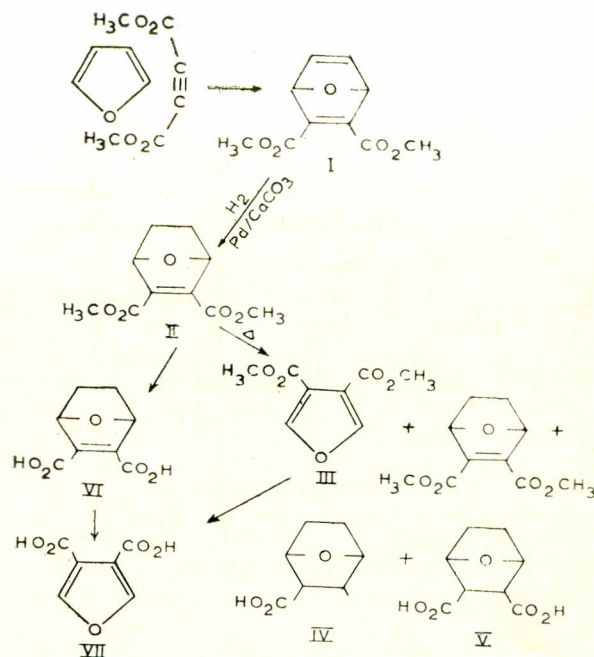
Chemistry Department, The University of Birmingham,
England

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During our investigations on the spectroscopic characteristics of various ring-substituted carbonyl derivatives of pyrrole, ^{1,3}furan, and thiophen convenient routes to difficultly accessible 3-acylfurans were investigated. In this connection we have discovered that furan-3,4-dicarboxylic acid can be prepared more conveniently and in high yields by a modification of the method of Alder and Rikert.⁴

Furan undergoes the Diels-Alder type of addition reactions, and Alder and Rikert⁴ have reported the addition of furan to ethyl acetylenedicarboxylate. The adduct can be hydrogenated preferentially at the isolated double bond and the reduced product on pyrolysis loses ethylene to give dimethyl furan-3,4-dicarboxylate (III). Diels *et al.*⁵ reported the same adduct by refluxing methyl acetylenedicarboxylate with furan in ether. Although the yields in these reactions have not been reported, the possibility of synthesizing furan-3,4-dicarboxylic acid by this method appeared promising.

Dimethyl acetylenedicarboxylate and furan were reacted in a sealed tube using the procedure of Alder and Rikert.⁴ The adduct, dimethyl 3,6-oxy-3,6-dihydrophthalate (I) was hydrogenated in ethyl acetate using Pd/CaCO₃ as a catalyst; under these conditions hydrogenation ceased after the absorption of one mole of hydrogen. The hydrogenated adduct (II) on distillation under reduced pressure gave, as the main fraction, a mixture of dimethyl furan-3,4-dicarboxylate (III) and the undecomposed adduct. However, at higher temperatures, a new fraction, not reported by previous workers, was obtained from which two crystalline compounds were isolated. These are probably the acids IV and V as inferred from their IR spectra and elemental analysis. The



combined yield of dimethyl furan-3,4-dicarboxylate and the undecomposed adduct was found to be low (38%) and no significant improvement was achieved by carrying out the initial condensation using the procedure of Diels *et al.*⁵ Moreover, the pyrolysis of the adduct was found to be uncertain and in one experiment the hydrogenated adduct distilled without any liberation of ethylene. These drawbacks led us to develop a modified synthetic procedure described below.

The crystalline adduct II was hydrolysed with aqueous sodium hydroxide and the corresponding 3,6-oxy-3,4,5,6-tetrahydrophthalic acid (VI) was obtained in 96% yield. Liberation of ethylene from VI by heating with quinoline at 170° gave furan-3,4-dicarboxylic acid (VII) in 85% yield.

Experimental

Dimethyl 3,6-oxy-3,4,5,6-tetrahydrophthalate (II).—Dimethyl acetylenedicarboxylate (14 g) and furan (7 g) were heated in a sealed tube at 100° for 18 hr. The crude adduct was hydrogenated in ethyl acetate (100/ml) using Pd-CaCO₃ (250mg). The catalyst was removed by filtration and the filtrate on recovering the solvent gave II (20.2 g) as a viscous oil.

*Present address: Chemical Research Division, Central Laboratories, PCSIR, Karachi.

Pyrolysis of II at 10 mm gave the following three fractions:

Fraction I. had b.p. 130-140° (4.7 g) and was hydrolysed with potassium hydroxide (2 g) in methanol (30 ml) for 5 hr. Acidification of the mixture and subsequent extraction with ether (4×50 ml) gave furan-3,4-dicarboxylic acid (1.8 g). Recrystallization from ethyl acetate afforded pure colourless needles, m.p. 212° lit. mp 212°.

Fraction II. had b.p., 140-165 (3 g) and on hydrolysis in a similar manner with methanolic KOH afforded furan-3,4-dicarboxylic acid (1.2 g).

Fraction III. had b.p. 165-180° (1.2 g) was recrystallised from light petroleum ethyl acetate to yield a mixture of two compounds m.p. 135-150°. Extraction with hot petrol (b.p. 40-60°) gave colourless needles of 3,6-oxy-1,2,3,4,5,6-hexahydrobenzoic acid (IV), m.p. 76-77°, (0.4 g) Found: C, 59.3; H, 6.9; C₇H₁₀O₃, requires C, 59.1; H, 7.0%; $\nu_{C=O}$ (CS₂) 1725 cm⁻¹. The petrol-insoluble portion on recrystallisation from petrol-benzene gave colourless cubes of 3,6-oxy-1,2,3,4,5,6-hexahydrophthalic acid (V) m.p. 156-157. Found: C, 51.4; H, 5.0; C₈H₁₀O₅ requires C, 51.6; H, 5.4%.

3,6-Oxy-3,4,5,6 tetrahydrophthalic acid.—Dimethyl 3,6-oxy-3,4,5,6-tetrahydrophthalate was prepared from dimethyl acetylenedicarboxylate (14 g) and furan (7 g) by heating them in a sealed tube at 100° for 18 hr and subsequent hydrogenation. Extraction of the adduct with hot petroleum (6×25 ml) afforded on cooling colourless needles of dimethyl 3,6-oxy-3,4,5,6-tetrahydrophthalate (II), (9.3 g) m.p. 52°. N.M.R. in CCl₄ showed bands at $\tau=6.2$ (CH₃), and $\tau=4.85$ (CH).

Hydrolysis of the ester with aqueous sodium hydroxide and subsequent acidification and extraction with ether gave 3,6-oxy-3,4,5,6-tetrahydrophthalic acid (VI), (7 g), m.p. 165-167°, lit. 4 m.p. 165°.

Furan-3,4-dicarboxylic acid.—The dicarboxylic acid (VI) (5 g) was heated in quinoline (10 ml) at 170° for 1.5 hr. The mixture was cooled and ether (50 ml) was added. Excess quinoline was removed by adding HCl (50%; 20 ml). The aqueous phase on extraction with ether and subsequent evaporation of the solvent gave furan-3,4-dicarboxylic acid (4.5 g) m.p. 212°; undepressed on admixture with the authentic sample. $\nu_{C=O}$ (CHCl₃) 1734 and 1742 Cm.⁻¹

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and the Microanalytical Department of the Birmingham University for carrying out elemental analysis. One of us (M.K.A.K.) thanks P.C.S.I.R., for the grant of study leave.

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CHEMICAL COMPOSITION OF ADHATODA VASICA LINN—II

M. EHSANUL HUQ, M. IKRAM AND S.A. WARSII

*North Regional Laboratories, Pakistan Council of
Scientific and Industrial Research, Peshawar*

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The isolation of vasicine and a new alkaloid vasicinine alongwith betaine has already been reported from the flowering tops of *Adhatoda vasica* in a previous communication.¹ An examination of the leaves, stems, roots and flowers was subsequently taken up to ascertain the amount of vasicine and vasicinine present in these parts. Vasicinine was found to be present in the stems, roots and flowers but could not be isolated from the leaves. Betaine could, however, be detected in the aqueous mother liquor and was precipitated out as the bismuth iodide complex from all parts of the plant.

The non-alkaloidal, ether-soluble fraction on subjecting to column chromatography yielded two crystalline compounds, one m.p. 140-41° and the other m.p. 61-62° respectively. The first melting at 140-41° was identified as β -sitosterol. β -Sitosterol was found to be present, in the flowering tops, flowers, and leaves but could not be detected in the roots or stems.

The second compound m.p. 62-63° was identified as tritriacontane (C₃₃H₆₆) a saturated hydrocarbon. The tritriacontane was present throughout the plant and it was possible to separate from the ether-soluble fraction of the main extract.

Experimental

In a fresh working, the plant material collected in March from Khyber Agency, West Pakistan, was separated into the roots, stems, leaves, flowering tops and flowers. Each part was separately extracted with ethyl alcohol (95%) in the manner described in the earlier paper.¹ The crude extract after the removal of the solvent was repeatedly extracted with ether. The dark green semisolid mass obtained on removal of the solvent was adsorbed on a column of activated alumina (May and Baker, Chromatographic alumina) and eluted with ether, acetone and chloroform.

The working of the flowering tops only is being described in detail. The ether soluble fraction of the crude extract (50 ml) from the flowering tops (1 kg) was put on a column of alumina (24×4 cm) and eluted with ether, acetone and chloroform.

Ether-soluble Fraction.—Six fractions of (40 ml) were collected. Each fraction was separately treated with activated charcoal, dried (Na₂SO₄), filtered and the solvent removed. The last fraction consisted of an oily liquid from which no crystalline compound could be separated. The first five fractions, on removal of the solvent and keeping in the cold, gave colourless crystalline compound which on recrystallisation from acetone finally melted at 140–41° (0.03%) and was identified as β -sitosterol.

The m.p. of the compound was undepressed on admixture with an authentic sample (recorded m.p. 140°²). The acetyl and benzoyl derivatives prepared according to the conventional methods were found to be melting at 120–27° (lit.² 127°) and 143–44° (lit.³ 144.5°) respectively. IR spectra of the isolated β -sitosterol corresponded with that of an authentic sample.

The oily fractions were not investigated further.

Acetone-soluble Fraction.—Six fractions (50 ml) were collected. The first two fractions on removal of the solvent were found to contain oil and were not investigated further. The last four fractions, deep brown in colour, on concentration and keeping in the cold deposited shining crystalline plates, m.p. 55–60°. On repeated crystallisation from a mixture of pet. ether and acetone (1:3), shining scaly crystals of constant m.p. 62–63° were obtained. Its m. p. was undepressed on admixture with an authentic sample m.p. 64–65°. The IR spectra of the compound and the authentic sample were superimposable.

Chloroform-soluble Fraction.—Three fractions of (50 ml) were collected. Treatment with activated charcoal and removal of the solvent did not give any solid crystalline product.

The greenish eluate from the roots, stems and leaves gave tritriacontane only. No β -sitosterol could be detected in the roots and stems of the plant.

Table I gives the amount of the alkaloidal and non-alkaloidal constituents present in the different parts of the plant:

TABLE I.

Plants	% Vasicine;	% Vasicine	% β - Sitos- terol	Tritri- acont- ane
Roots	0.011	0.04	nil	0.07
Stems	0.013	0.02	nil	0.06
Leaves	1.67	nil	0.013	0.075
Flowering tops	0.64	0.68	0.035	0.09
Flowers	0.15	0.35	0.03	0.07

Acknowledgement.—The authors are thankful to Dr. A. Khaliq of East Regional Laboratories, Dacca, for taking the IR spectra of tritriacontane and identifying it. Thanks are also due to Mr. Mohammad Iqbal Tahirkheli and Mr. Mohammad Nazir for their help in experimental work.

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TERMITE REARING AND TESTING OF MAKROLIN AS A WOOD-PRESERVATIVE AGAINST TERMITES. (ISOPTERA-TERMITIDAE).

MOHAMMAD WAHAJUDDIN ROOMI, I.A. KHAN
AND SHAHID H. AHSRAFI

Pesticide Research Section, Central Laboratories, Pakistan Council of Scientific and Industrial Research, Karachi

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A number of studies about termites have already been carried out, specially from the damage point

of view produced in wood of various kinds of buildings. They cause a loss estimated at 100 million dollars each year in the United States alone. Ninety-five per cent of termite damage in wood is caused by the subterranean type which includes the family *Termitidae* and whose members are commonly found in enormous number in Pakistan. Damage to metal and other types of impenetrable material is caused by the corrosive action of moisture brought up by the subterranean termites with the earth and excreted wood from which they build sheltered runways in them.

The present work deals with rearing of termites and the insecticidal evaluation of Makrolin against termite attack. Makrolin² is a new pesticide developed in these Laboratories.

Rearing of Termites.—The container described by Borrer and DeLong⁴ was slightly modified in size, and Perspex sheet was used in place of glass sheets. The container was fixed on the top of a box made of aluminium in the size of 3'×2'×6". The aluminium box acted as the base for general termite colony (Fig. 1). An observatory Perspex sheet was fixed in the lid for examining the development of the termite culture inside the box. Six Perspex sheet containers, 13"×13"×3", were fixed on the lid of the main box (Fig. 2). Each container had three holes at the bottom, coinciding with another three holes in the lid of the main box. The box was filled with the soil mixed with chips of "partal" wood and a few crushed yeast tablets for the development of the fungus gardens in the termite-colony. The six containers were half-filled with soil mixed with 0.1% yeast. Before closing the lid, the primary reproductives which

are essential for the foundation of new colonies were introduced in the box, together with moist infested wooden pieces, and a little water was sprinkled over the soil in the box. A few termites were also introduced into each container and a cotton pad soaked with water was kept inside each container. The eggs were normally deposited singly. The incubation period lasted for 30 to 35 days and the nymphal instars started appearing in enormous number. Within 3 months more than a million individuals were developed from the primary reproductives. The individuals produced in the early stages of the colony were of sterile castes namely workers and soldiers. The dead bodies of the termites were also eaten up by the individuals, which thus brought about continuous supply of fungus and symbiotic protozoan⁵ to the other members of the colony.

Makrolin as Wood Preservative.—In order to test Makrolin as wood-preservative against termite attack, the "partal" wood which is commonly available in Pakistan, was selected. In a preliminary experiment in which wooden pickets were treated with 25% Makrolin, the control was eaten up by the termites after 95 days, but the treated wood was not attacked by the termites. Then 40 wooden pickets of small size and shape were used for experimentation. At the head of each picket an aluminium plate was attached over which a number was punched serially. Different concentrations of Makrolin were prepared ranging from 5% to 35% in kerosene as solvent. Five pickets were dipped for 24 hr in each jar containing 5%, 10%, 15%, 20%, 25%, 30%, and 35% Makrolin. For control five pickets were treated with kerosene only. The pickets were

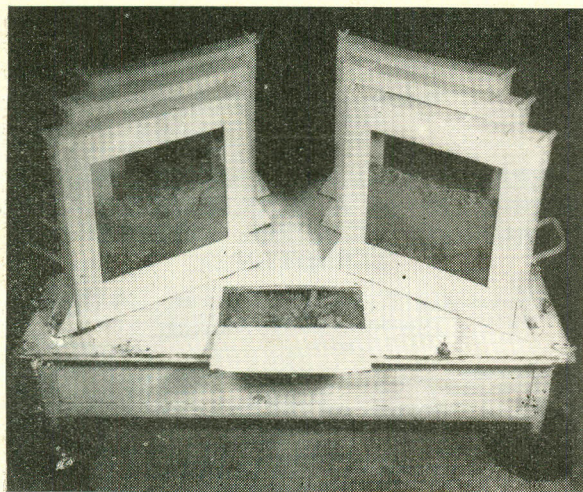


Fig. 1.—Main termite culture-box with six perspex-sheet type of containers.

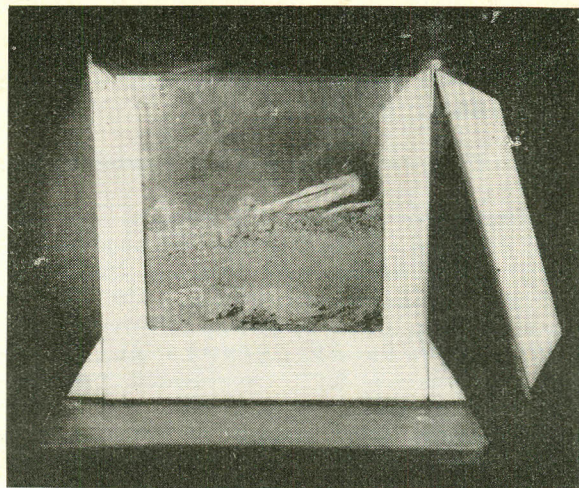


Fig. 2.—Enlargement of a single perspex-sheet type of containers.

then taken out and dried in air for 48 hrs. These pickets were buried in termite-infested areas and inspected periodically at an interval of seven days.

The treated pickets were finally taken out from the termite infested soil after one year, and the control after two months. The control was severely damaged by the termites within such a short period (Fig. 3), while the treated pickets were not attacked by them (Fig. 4). Even the 5% solution of Makrolin was found quite satisfactory to keep away the termites from the wood.



Fig. 3.—Control destroyed severely by the termites.

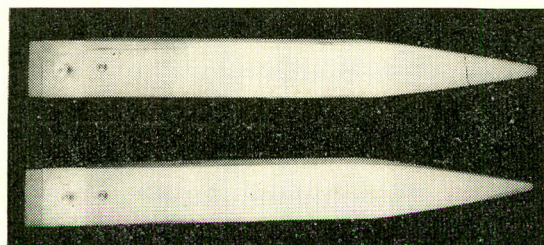


Fig. 4.—Treated wooden-pickets with makrolin showing no attack of termites on them.

The length of time over which Makrolin remains effective depends on a number of factors, including soil type, exposure to weather and the kind of termite involved. The field trials were carried out at Tandojam, West Pakistan, in dry fields. It was also interesting to note that when the wood is treated with Makrolin it always gets fixed into the wood cellulose and the treated wood gives the smell of Makrolin as long as three years after the treatment.

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THE HEALTHY JUICE EFFECT IN VIRUS TRANSMISSION*

SORAYA AZEEM

Department of Botany, University of Karachi

C.E. YARWOOD

Department of Plant Pathology, University of California, Berkeley

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Introduction

Many if not most virus-infected tissues contain inhibitors of virus infection.¹ With the two viruses studied here, the addition of Bentonite to the inoculum may greatly increase virus infection, and the effect of Bentonite was tentatively attributed to its effect in inactivating inhibitors.² In the present study it is found that the addition of healthy plant juice to virus: Bentonite: K_2HPO_4 inoculum greatly increases its infectivity.

Methods

Inoculum of tomato ringspot virus (TRSV) was taken from systemically-infected *Erigeron glaucus*,² and assayed on primary leaves of Pinto bean.

Inoculum of cucumber mosaic virus was taken from locally-infected cucumber cotyledons and assayed on cowpea primary leaves, bean primary leaves and cucumber cotyledons. Inoculum was prepared by grinding 0.1 g donor tissue and 0.1 g commercial dry bentonite powder in 1 ml 1%

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K_2HPO_4 , and then diluting to 10 ml with further K_2HPO_4 . If healthy leaf tissue was used, it was added to the inoculum before grinding. The final inoculum thus contained 1% donor tissue, 1% Bentonite, and 1% K_2HPO_4 , with or without 1% healthy tissue. The healthy tissues used were primary cowpea leaves, cucumber cotyledons or secondary beet leaves. Inoculum was applied by dipping cotton swabs (Q-tips) into the inoculum and rubbing them over the surface of the indicator plants. Leaves were quick dried after inoculation. Lesions were counted at about 7 days after inoculation for TRSV and 3 days after inoculation for CMV.

Results

Results are presented in Table 1. The addition of healthy tissue increase virus transmission in 13 out of 14 cases. The greatest increase, 750-fold, was for healthy sugar beet tissue added to CMV and assayed on cowpea. The average increase due to healthy tissue was 50-fold.

The results given in Table 1 are the average of more than 10 replications of trials.

TABLE 1.—EFFECT OF BENTONITE AND HEALTHY PLANT TISSUE ON TRANSMISSION OF TOMATO RINGSPOT VIRUS AND CUCUMBER MOSAIC VIRUS.

Inoculum	Indicator host	1% Bentonite	Healthy tissue and numbers of assay lesions per leaf			
			0	Cowpea	Cucumber	Beet
TRSV	Cowpea	0	0	3	3	2
		+	0	15	22	15
CMV	Cowpea	0	191	182	102	4
		+	798	1003	163	1458
	Cucumber	0	9	25	6	1
		+	22	17	32	36
	Bean	0	21	12	4	22
		+	25	196	122	171

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SYNTHESIS OF 3-METHYL-4-BENZYLIDENE ISOXAZOLONE AND 4,4-BENZAL-BIS-(3-METHYL-ISOXAZOLONE-5)

A. EHSAN, SHAUKAT ALI, IFTIKHAR AHMED

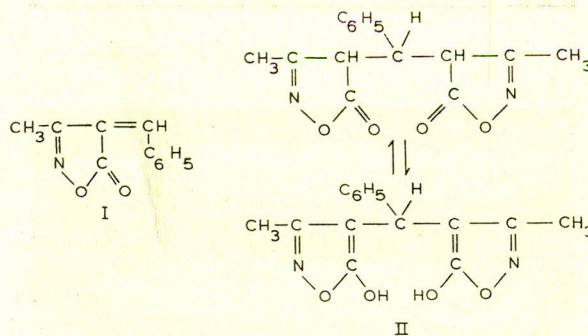
AND

KARIMULLAH*

West Regional Laboratories, Pakistan Council of Scientific and Industrial Research, Lahore

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Compounds I and II have been reported.¹⁻³ Compound II has been obtained by treating I with hot aqueous 5% sodium carbonate or sodium hydroxide.



We have obtained II in pure form by simply mixing ethyl acetoacetate and benzaldehyde in hydroxylamine hydrochloride solution in water, and leaving the reaction mixture overnight at room temperature. A cream coloured crystalline compound separated out. This compound is identical with that obtained by Donleavy and Gilbert³ from I. The IR spectrum of II corresponds with that of an authentic sample.

Bis-isoxazolone II is insoluble in carbon tetrachloride, carbon disulphide and is insufficiently soluble in deuteriochloroform. It is, however, soluble in polar solvents like methanol, pyridine, acetone, dimethyl sulphoxide and trifluoroacetic acid. In dimethyl sulphoxide solution the 60 MC NMR spectrum displayed bands at 1.8 ppm (6H, singlet, methyl protons), 4.82 ppm (1H, singlet, methine proton adjacent to phenyl) and 7.25-7.4 and 8.15 ppm (5H, multiplet, phenyl protons). A broad band (2H, enolic OH protons) appeared downfield below 8.0 ppm. In trifluoroacetic acid compound II gave signals at 2.2 ppm (6H, singlet,

*Now at the Defence Science Organisation Cell, Lahore.

methyl protons), 5.22 ppm (1H, singlet, methine proton adjacent to phenyl) and 7.3 to 8.0 ppm (5H, multiplets, phenyl). The methine proton appeared as a sharp singlet in pyridine solution as well. It is, therefore, inferred that II exists predominantly in the 5-OH form. This was further verified by exchange with D₂O in dimethyl sulphoxide solution.

Compound II behaves as a carboxylic acid (100%) on titration with alkali and reacts quantitatively with diphenyl diazomethane. The kinetics of this reaction is also under investigation. These properties are in line with the observation of Boulton and Katritzky.⁴

Donleavy and Gilbert³ observed that II turned pink and then yellow on exposure to air, but the compound II prepared as above does not behave similarly. However, it shows the reported colour change if it is dissolved in 5% sodium carbonate and reprecipitated with dilute acid.

Compound I is soluble in deuteriochloroform and the 60 MC NMR spectrum displayed bands at 2.28 ppm (3H, singlet, methyl protons), 7.23 ppm (1H, singlet, olefinic proton) and 7.5 and 8.3 ppm (5H, multiplets, phenyl protons).

The physical characteristics of the products obtained from formaldehyde, acetaldehyde, propionaldehyde and salicylaldehyde indicate that they behave in a manner similar to benzaldehyde, whereas anisaldehyde, cinnamaldehyde and vanillin give only isoxazolone of type I under any set of conditions. Behaviour of other aromatic and aliphatic aldehydes is also under investigation. Alkylidenebis-3-methylisoxazolone-5 decomposes in the presence of uranium, palladium and copper

salts. A method has been developed for the quantitative estimation of uranium and palladium.

Experimental

Bisoxazolone II.—Ethyl acetoacetate (0.05 mole) and benzaldehyde (0.025 mole) were mixed in 0.5% hydroxylamine hydrochloride solution in water (1000 ml) and the mixture left overnight at below 25°. Only II separated out in 34% yield, m.p. 150-152° dec., lit.³ m.p. 150-151°. Found: C, 63.31; H, 4.75; N, 9.61. Calculated for C₁₅H₁₄N₂O₄: C, 62.93; H, 4.93; N, 9.79.

Isoxazolone I.—Ethyl acetoacetate (0.05 mole) and 0.5% hydroxylamine hydrochloride solution in water (1000 ml) were allowed to stand overnight at room temperature and then benzaldehyde (0.05 mole) was added. After a few minutes, I separated out in 73% yield, m.p. 142-144°, lit.³ m.p. 146-147°. Found: C, 70.10; H, 4.75; N, 7.60. Calculated for C₁₁H₉NO₂: C, 70.58; H, 4.85; N, 7.48.

Acknowledgement.—We are indebted to Drs. K.J. Toyne and N.A. Mirza for running the NMR spectra.

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BOOK NOTICES

Industrial Crystalization. A.W. Bamforth.—361 pp. Leonard Hill, London, 1965. Price 75s.

Written for the manufacturers of chemicals, plant buyers and operators this book will also be invaluable to students. It contains descriptions and operating data on a wide range of crystallization plant and immediate auxiliaries. Its coverage is more comprehensive than any previously published work, for it includes in one volume, descriptions of leading equipment from many countries. Data, flowsheets and much other information have been

contributed by crystal producers and by many companies who design apparatus. Of great value to those concerned with crystallization plant is the coverage given to ancilliary equipment such as pumps, vacuum equipment and centrifuges.

Textile Finishing. A. J. Hall, 453 pp. Heywood Books, London, 1952. Price 105s.

The first two editions of this book were published under the title 'A Handbook of Textile Finishing'. When revisions were being made