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Study of the reaction of N,N-diethylhydrazine with nitrobenzoic acids

U Ch Akhmedov*[a], H S Tadjimukhamedov[b], A K Abdushukurov[b], B Tashxadjayev[c], K K Turgunov[c] & A K Nomozov[d,e]

[a] Termez State University, Termez, 190100, Uzbekistan

[b] National University of Uzbekistan named after Mirzo Ulugbek, 100174, Tashkent, Uzbekistan

[c] Institute of Chemistry of Plant Substances, Termez, 190100, Uzbekistan

[d] Department of Chemical Engineering, Termez State University of Engineering and Agrotechnologies, Termez 190111, Uzbekistan

[e] Department of Medical and Biological Chemistry, Termez Branch of Tashkent Medical University, Termez 190111, Uzbekistan

E-mail: akhmedov@tdmau.uz

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In this study, the synthesis of the N,N-diethylhydrazine salt of nitrobenzoic acid isomers containing an electron-withdrawing group in the molecule has been carried out, and it is found that the type of solvents, molar ratios of the starting materials, and reaction time parameters in the preparation of this N,N-diethylhydrazine salt affects the product yield. According to the results, when parameters such as molar ratios of the starting materials, solvents, temperature, and reaction time are studied, the reaction yield increases to 90%. The structure of this synthesized compound has been determined using IR spectroscopy, ^{1}H NMR, and X-ray analysis.

Keywords: N,N-Diethylhydrazine, Nitrobenzoic acid isomers, Quaternary ammonium salts, X-ray structural analysis, Ion-molecular complex

Hydrazine and its substituted derivatives are highly reactive compounds, which can be used to synthesize various substances with new properties. Biologically active substances that are widely used in agriculture[1] and medicine[2,3] have been found in a series of hydrazine derivatives. There are data in the literature on obtaining oxalic acid salts with dimethyl-, diethyl-, di-n-propyl-, di-n-butylhydrazines[4], as well as hydrazides in the formula $RCONHNMe_2$, where R=C_6H_5, 4-CH_3-C_6H_4, 4-NO_2-C_6H_4)[5-8]. However, they do not provide data on reaction conditions and product yields. This study revealed that unactivated peroxymonosulfate (PMS) oxidation can lead to the formation of N-nitrosodimethylamine (NDMA), a suspected human carcinogen, in water containing N,N-dimethylhydrazine derivatives. Significant NDMA formation was observed from three compounds under PMS treatment. Among them, 1,1,1',1'-tetramethyl-4,4'-(methylene-di-p-phenylene) disemicarbazide (TMDS), an industrial light stabilizer, was selected to investigate oxidation kinetics, transformation pathways, and NDMA formation mechanisms[9,10]. NDMA forms efficiently during ozonation of dimethylhydrazine-based compounds like DMZ and 2-F-DMH at *p*H 7. Using O_3 with PMS reduced NDMA formation more effectively (50–65%) than with H_2O_2 (10–25%) at an 8:1 oxidant-to-ozone ratio. This is due to sulfate radicals (SO_4^{2-}), which showed a strong correlation with reduced NDMA. However, ozone reacts rapidly with the precursors, limiting the effect of PMS or H_2O_2. Applying ozone in smaller, repeated doses further lowered NDMA levels. Bromate formation was higher with O_3/PMS, highlighting the need to monitor both NDMA and bromate in treatment systems[10].

The release of the highly toxic rocket fuel 1,1-dimethylhydrazine (UDMH) and its transformation products poses significant environmental and human health risks. This study provides the first data on the spatial distribution and quantification of UDMH and its major transformation products in peat bog soil near a subarctic rocket fall site. One hundred soil samples and one surface water sample were analyzed using a previously developed methodology. High concentrations of UDMH and its transformation products were found within 10 meters of the impact site[11]. The reaction of 1,1-diphenylhydrazine with $Ti(NMe_2)_2Cl_2$ yielded the monomeric titanium hydrazido(2-) complex $Ti(NNPh_2)Cl_2(HNMe_2)_2$ (1) in

$$2(C_2H_5)_2NH + 2NaNO_2 + H_2SO_4 \longrightarrow 2(C_2H_5)_2NNO + Na_2SO_4 + 2H_2O$$

$$(C_2H_5)_2NNO + 4\,[H] \xrightarrow{Zn/Hg + HCl} (C_2H_5)_2NNH_2 + H_2O$$

Scheme 1 — Synthesis of N,N-diethylhydrazine

nearly quantitative yield. When $Ti(NMe_2)_2Cl_2$ reacted with less sterically bulky ligands like 1,1-dimethylhydrazine or N-aminopiperidine, dimeric mu-eta²,eta¹-bridged compounds were formed: Ti_2(mu-eta²,eta¹-$NNMe_2)_2Cl_4(HNMe_2)_2$ (2) and Ti_2[mu-eta²,eta¹-$NN(CH_2)_5]_2Cl_4(HNMe_2)_3$ (3)[12,13].

The main aim of this study is to synthesize and determine the structure of complexes of N,N-diethylhydrazine with *o*-, *m*-, and *p*-nitrobenzoic acids.

Methods

NMR-analysis

^{1}H NMR spectra were obtained on a Varian XL-100/15 spectrometer with an operating frequency of 100 MHz in the slow-pass mode, internal standard - HMDS, the accuracy of chemical shift determination is ±1 ppm in D_2O solution.

IR-analysis

IR spectra were recorded on a Specord 75IR spectrophotometer in potassium bromide tablets in the range of 400-4000 cm^{-1}.

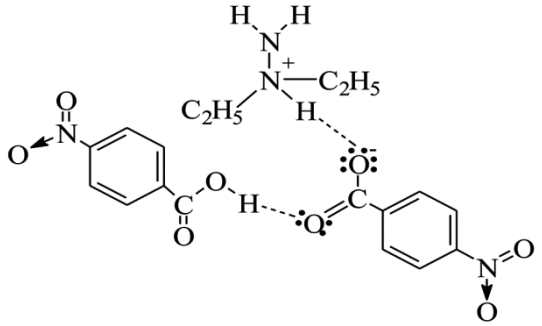
X-ray structural analysis

Crystallographic parameters of single crystals were determined and refined on a CCD diffractometer «Xcalibur Oxford Diffraction» (CuK-radiation, graphite monochromator, at RT). The experimental data collection strategy was implemented using the CrysAlisPro program.

Experimental Section

In order to study the reaction of N,N-diethylhydrazine (N,N-DEH) with isomers of nitrobenzoic acids, N,N-diethylhydrazine was synthesized using known methods in two stages: nitrosation of diethylamine and reduction of N-nitrosodiethylamine. The reaction is depicted in Scheme 1.

In order to study the quaternization reactions of the synthesized N,N-DEH with *o*-, *m*-, *p*-nitrobenzoic acids, the reactions were carried out in abs. ethanol, in a 1:1 molar ratio, by boiling for 3 hours, the corresponding nitrobenzoate salts were obtained in yields of 30, 35 and 32%. It was found that increasing



Scheme 2 — Ionic-molecular complex of *p*-nitrobenzoic acid with N,N-DEH

the reaction time to 6 hours did not significantly affect the product yield. When these reactions were carried out in benzene, the product yields were 43, 50 and 48%. When the amount of N,N-DEH was increased to 1.2 mol relative to 1 mol of acid, an increase in product yields of 36, 41 and 39% was observed. The results of the conducted experiment showed that the state of the electron-acceptor nitro group in nitrobenzoic acids does not significantly affect the product yield under the conditions studied.

When the single crystal structure of the corresponding product of N,N-DEH with *p*-nitrobenzoic acid was studied by X-ray diffraction (XRD), it was found that this compound has an ionic-molecular complex structure consisting of one molecule of N,N-DEH and two molecules of *p*-nitrobenzoic acid (Scheme 2). During the formation of the complex, the first acid molecule donates its proton to the nitrogen atom holding the ethyl groups of N,N-DEH, forming a salt, and the second acid molecule forms a strong hydrogen bond with the carbonyl oxygen of the complex.

Considering that the resulting ionic-molecular complex has a 1:2 ratio, the reaction of N,N-DEH with *p*-nitrobenzoic acid in a 1:2 molar ratio was carried out in benzene under heating for 3 hours, resulting in a 90% yield.

Given the high yield of the product obtained in benzene, in order to study the effect of reaction time and molar ratios of reagents on the yield of the reactions of N,N-DEH with isomeric nitrobenzoic acids, we carried out the reactions in benzene for various times, using the example of *p*-nitrobenzoic

acid. Alternative conditions for these reactions were heating in benzene for 3 hours with a molar ratio of reagents of 1.2:2 (Table 1).

Under the specified alternative conditions, the reactions were carried out with 81% yield of o-nitrobenzoic acid (I), 87% yield of *m*-nitrobenzoic acid (II), and 97% yield of *p*-nitrobenzoic acid (III). The reaction equation is shown in Scheme 3.

Complex of *o*-Nitrobenzoic acid with N,N-diethylhydrazinium *o*-nitrobenzoate, I

A solution of 1.05 g (0.012 mol) of N,N-DEH in 15 mL of abs. benzene and 3.34 g (0.02 mol) of *o*-nitrobenzoic acid in 15 mL of abs. benzene were placed in a round-bottomed flask equipped with a reflux condenser and boiled at 70-80°C for 3 hours. The benzene was evaporated, and the liquid portion was dried in a desiccator with $CaCl_2$. The resulting crystals were recrystallized from abs. benzene. The yield of the product was 3.41 g (81%). I- yellowish crystalline substance. T_{liquid}=64-65°C, (abs. benzene). Found: C, 51.22; H, 5.73; N, 12.99. Calcd for $C_{18}H_{22}N_4O_8$: C, 51.18; H, 5.21; N, 13.27%.

Complex of *m*-Nitrobenzoic acid with N,N-diethylhydrazinium *m*-nitrobenzoate, II

In the above method, a solution of 1.05 g (0.012 mol) of N,N-DEH in 15 mL of abs. benzene was reacted with a solution of 3.34 g (0.02 mol) of m-nitrobenzoic acid in 15 mL of abs. benzene to obtain 3.67 g (87%) of the product. II- yellowish crystalline substance. T_{liquid}= 71-72°C, (abs. benzene). Found: C, 51.53; H, 5.96; N, 12.30. Calcd for $C_{18}H_{22}N_4O_8$: C, 51.18; H, 5.21; N, 13.27%.

Complex of *p*-Nitrobenzoic acid with N,N-diethylhydrazinium *p*-nitrobenzoate, III

In the above method, a solution of 1.05 g (0.012 mol) of N,N-DEH in 15 mL of abs. benzene was reacted with a solution of 3.34 g (0.02 mol) of p-nitrobenzoic acid in 15 mL of abs. benzene to obtain 4.1 g (97%) of the product. III-yellow crystalline substance. T_{liquid}=139-140°C (abs. benzene). Found: C, 51.76; H, 5.83; N, 13.32. Calcd for $C_{18}H_{22}N_4O_8$: C, 51.18; H, 5.21; N, 13.27%.

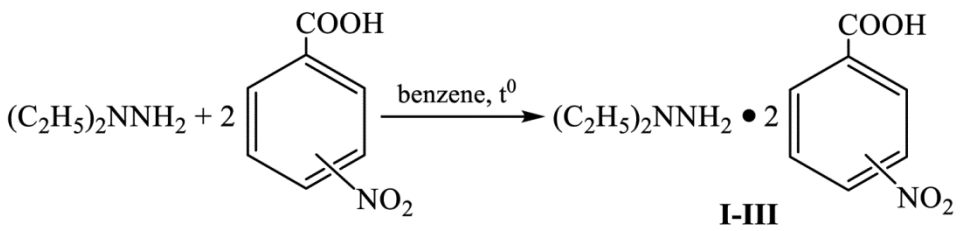
X-ray analysis

The chemical and spectral characteristics of the obtained substance do not allow us to unambiguously establish the structure of the ion-molecular complex, *i.e.* they do not determine the nature of its salt formation in terms of the formation of inter- and intramolecular hydrogen bonds. For this reason, single crystals of N,N-diethylhydrazinium (III) p-nitrobenzoate were obtained and subjected to X-ray diffraction analysis (XRD).

From the XRD analysis it follows that the substance is a salt complex consisting of one molecule of protonated N,N-DEH (cation) and two molecules of *p*-nitrobenzoic acid, where one of them is a molecular ion (anion). Fig. 1 shows the spatial structure of this complex salt (independent part of the unit cell). It should be noted that the structure of crystal III exhibits statistical disorder in the arrangement of protonated N,N-DEH: two methylene groups (atoms C9 and C10) and the NH_2 group occupy two equivalent positions (with a multiplicity of 0.5), and the methyl carbon atoms (atoms C8, C11) and the N1 atom are fixed with a multiplicity of 1. Consequently, the hydrogen atoms of the terminal methyl groups are also statistically disordered. The

Table 1 — Effect of reactant molar ratios and reaction duration on the yield of the reaction of N,N-DEH with *p*-nitrobenzoic acid (reaction temperature 70-80°C, solvent - benzene)

Mole ratios of reagents: $(C_2H_5)_2NNH_2$: *p*-$N0_2C_6H_4COOH$	Duration of reaction (h)	Product yield (%)
1: 2	1	78
1: 2	2	86
1: 2	3	90
1: 2	4	90
1: 2	5	91
1: 2	6	92
1,1: 2	3	95
1,2: 2	3	97
1,3: 2	3	97
1,4: 2	3	97



Scheme 3 — Complex of *o*-, *p*-,*m*-Nitrobenzoic acid

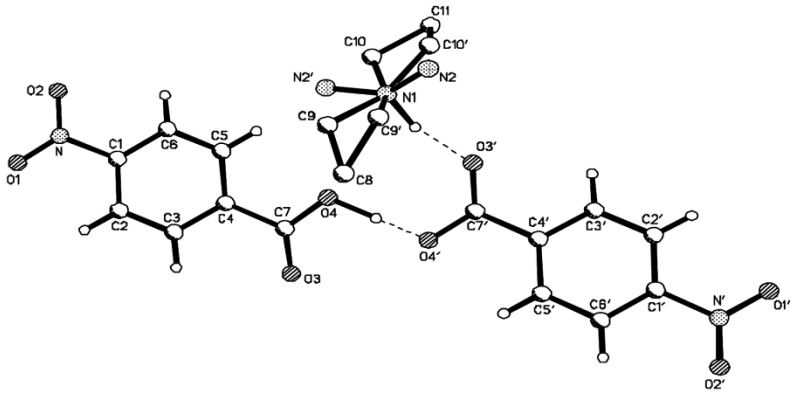


Fig. 1 — Spatial structure and numbering of atoms in salt complex III (hydrogen atoms of the "disordered" region of the diethylhydrazine anion are not shown).

ion molecular salt complex 1 is formed due to H-bonds of the O-H…O and N-H…O types. In the crystal, the molecules of *p*-nitrobenzoic acid with oxy groups are close together and form a strong H-bond, as evidenced by the O4…O4' distance (2.47 Å) and the almost mid-position of the bonding hydrogen atom H4 between the oxygen atoms O4 and O4' (the H4-O4 distance is 1.19 Å and the H4-O4' distance is 1.29 Å, and the O4-H4-O4' angle is 176.8°). Another H-bond is formed between the protonated N1 atom of diethylhydrazine and the oxygen atom of nitrobenzoic acid O3': the N1…O3' distance is 2.78, and the H1…O3' distance is 1.71 Å, and the N1-H1…O3' angle is 164.0°.

In the crystal, no other intermolecular interactions are observed; the formed salt complexes (skeletons) are located at van der Waals interaction distances. In the crystal, two independent molecules of p-nitrobenzoic acid are practically flat (with an accuracy of ±0.03 Å) and lie parallel, although the carboxyl groups in them are slightly rotated (14.10) relative to this plane. In the diethylhydrazine part of the complex, the protonated nitrogen atom has a distorted tetrahedral configuration.

The bond lengths and valence angles observed in salt complex III are given in Table 2. The bond lengths observed in aromatic rings and nitro groups (N=O) are identical within the experimental error and do not differ from the generally accepted values[14,15].

However, in the carboxyl part of two independently found molecules of *p*-nitrobenzoic acid, a strong difference in the lengths of the C-O bonds is observed, which varies from 1.214 to 1.292 Å. This phenomenon is explained by the different nature of the C-O bonds in complex III (see Fig. 1). The isolated double bond O3=C7, not participating in

Table 2 — Bond length (r, Å) and bond angles (w, deg.) in complex III

Bond	R	Angle	w
N-O(1)	1.217(3)	O(1)-N-O(2)	123.3(2)
N-O(2)	1.220(3)	O(1)-N-C(1)	118.9(2)
N-C(1)	1.467(3)	O(2)-N-C(1)	117.8(2)
O(3)-C(7)	1.214(3)	C(6)-C(1)-C(2)	122.5(2)
O(4)-C(7)	1.292(3)	C(6)-C(1)-N	119.1(2)
C(1)-C(6)	1.367(4)	C(2)-C(1)-N	118.4(2)
C(1)-C(2)	1.383(4)	C(1)-C(2)-C(3)	117.9(2)
C(2)-C(3)	1.386(3)	C(4)-C(3)-C(2)	120.7(2)
C(3)-C(4)	1.375(3)	C(3)-C(4)-C(5)	119.8(2)
C(4)-C(5)	1.387(3)	C(3)-C(4)-C(7)	118.9(2)
C(4)-C(7)	1.497(3)	C(5)-C(4)-C(7)	121.3(2)
C(5)-C(6)	1.378(3)	C(6)-C(5)-C(4)	120.4(2)
N'-O(1')	1.213(3)	C(1)-C(6)-C(5)	118.6(2)
N'-O(2')	1.215(3)	O(3)-C(7)-O(4)	124.6(2)
N'-C(1')	1.467(3)	O(3)-C(7)-C(4)	121.0(2)
O(3')-C(7')	1.232(3)	O(4)-C(7)-C(4)	114.4(2)
O(4')-C(7')	1.263(3)	O(1')-N'-O(2')	123.2(2)
C(1')-C(6')	1.373(3)	O(1')-N'-C(1')	118.7(2)
C(1')-C(2')	1.376(3)	O(2')-N'-C(1')	118.1(2)
C(2')-C(3')	1.384(3)	C(6')-C(1')-C(2')	122.6(2)
C(3')-C(4')	1.378(3)	C(6')-C(1')-N'	118.4(2)
C(4')-C(5')	1.387(3)	C(2')-C(1')-N'	118.9(2)
C(4')-C(7')	1.507(3)	C(1')-C(2')-C(3')	118.2(2)
C(5')-C(6')	1.386(3)	C(4')-C(3')-C(2')	120.9(2)
N(1)-C(9')	1.435(8)	C(3')-C(4')-C(5')	119.2(2)
N(1)-C(10)	1.439(8)	C(3')-C(4')-C(7')	120.3(2)
N(1)-N(2')	1.462(6)	C(5')-C(4')-C(7')	120.4(2)
N(1)-C(10')	1.466(8)	C(6')-C(5')-C(4')	120.9(2)
N(1)-N(2)	1.478(6)	C(1')-C(6')-C(5')	118.1(2)
N(1)-C(9)	1.502(8)	O(3')-C(7')-O(4')	125.4(2)
C(8)-C(9)	1.470(9)	O(3')-C(7')-C(4')	119.0(2)
C(8)-C(9')	1.484(9)	O(4')-C(7')-C(4')	115.7(2)
C(11)-C(10')	1.532(10)	C(9')-N(1)-N(2')	114.0(5)
C(11)-C(10)	1.548(11)	C(9')-N(1)-C(10')	116.8(5)
		N(2')-N(1)-C(10')	112.6(5)
		C(10)-N(1)-N(2)	113.2(5)
		C(10)-N(1)-C(9)	115.2(5)
		N(2)-N(1)-C(9)	111.4(5)
		C(8)-C(9)-N(1)	114.7(5)
		N(1)-C(10)-C(11)	113.6(5)
		N(1)-C(9')-C(8)	118.0(5)
		N(1)-C(10')-C(11)	113.0(5)

Table 3 — Specific absorption bands in the IR spectrum of compounds obtained as a result of the reactions of N,N-DEH with nitrobenzoic acid isomers, cm^{-1}

Type of substance	Free NH_2 (v)	N^+-H	COO^-(v)	C=O (v)	C-O (v)	C_2H_5 (v)	NO_2 (v)	Valence vibrations of the C=C bond in the aromatic ring	Deformation vibrations of a substituted benzene ring
I	3240, 3350	2480		1700		2975	1500, δ: 1340	1580	(1,2) $4\delta_{CH}$=730
II	3150, 3250	2260, 2580	1580		1050	2960	1550, δ: 1330	1500	(1.3) $3\delta_{CH}$=805 δ_{CH}=880
III	3230, 3330	2470		1690		2960	1500, δ: 1320	1585	(1,4) $2\delta_{CH}$=850

intermolecular interactions, takes on the value of 1.214(3) Å, which is characteristic of such bonds. The same bond O3'-C7', formed in another molecule – the residue of *p*-nitrobenzoic acid, is slightly elongated 1.232(3) Å, the reason for which is the participation of the O3' atom in the donor-acceptor interaction with the protonated N1 atom of diethylhydrazine. The lengthening of the O4-C7 distance (1.292(3) Å) compared to the same O4'-C7' (1.263(3) Å) and the location of the H atom near O4 (than O4') indicate that this molecule is more "neutral" - not participating in the protonation of diethylhydrazine[16,17].

IR and ^{1}H NMR analysis

In the IR spectra of compounds obtained from the reaction of N,N-DEH with carboxylic acids, absorptions of the valence vibrations of the N+H bond specific to the nitrogen atom to which the acid proton is attached at 2000-2759 and of the free NH_2 group to which the acid proton is not attached at 3140-3462 were observed. The IR spectral data of compounds obtained from the reaction of N,N-DEH with some carboxylic acids are presented in Table 3, and the ^{1}H NMR spectral data are presented in Table 4.

X-ray structural analysis

Monocrystalline III obtained by recrystallization from heptane at RT, crystalline transparent, have the form of drawn out prism. The parameters of the elementary cell and the intensity of radiation are determined by the four-ring diffractometer STOE Stadi-4 (θ/2θ-scanning) using MoK_α (graphite monochromator). Repairs and payments are not made. Basic crystallographic data and conditions for the implementation of X-ray structural experiments are given in Table 5.

The structure was solved by the direct method using the SHELXS-97 software package. The structure was refined by the least squares method (LSM) in the full-matrix isotropic-anisotropic approximation using the SHELXL-97 program. All non-hydrogen atoms were refined by the full-matrix LSM (by F2) in the anisotropic approximation. The hydrogen atom positions were revealed geometrically and refined with fixed isotropic displacement parameters Uiso=nUeq, where n=1.5 for methyl

Table 4 — ^{1}H NMR-spectral data of compounds obtained as a result of the reactions of N,N-DEG with nitrobenzoic acid isomers, m.a.

Type of substance	CH_3 (C_2H_5)	CH_2 (C_2H_5)	Aromatic ring protons
I	1,15-1,30 т	3,14-3,33 к	7,53-7,80 м
II	1,16-1,29 т	3,10-3,30 к	7,50-8,64 м
III	1,0-1,20 т	2,86-3,10 к	7,90-8,20 м

Table 5 — Crystallographic data, characteristic of the experiment and parameters of structure III

Empirical formula	$C_{18}H_{22}N_4O_8$
Molecular weight	422.40
Temperature, K	293
Spatial group	P-1, Z=2
a, Å	8.063(2)
b, Å	11.532(2)
c, Å	11.934(2)
A	113.00(3)
B	97.39(3)
Γ	95.61(3)
V, $Å^3$	999.6(3)
ρ, $г/см^3$	1.403
Absorption coefficient., μ (Mo)	0.112(3)
Crystal size, mm	1.00x0.75×0.6 mm^3
Range of angles θ, deg.	From 1.89 to 24.99
Total number of reflections	3523
Number of reflections [I>2σ(I)]	2511
R-factor [I>2σ(I)]	R1=0.0639, wR^2=0.1891
R-factor (over the entire array)	R1=0.0859, wR^2=0. 0.2158
GOOF	1.069
Electron density difference peaks	0.27 and –0.17 $eÅ^{-3}$

groups and 1.2 for the rest, and Ueq is the equivalent isotropic displacement parameter of the corresponding carbon atoms. The hydrogen atom of the hydroxyl group was found from the difference synthesis of electron density and refined isotropically[18,19].

The X-ray diffraction data were deposited as a CIF file at the Cambridge Crystallographic Data Centre (CCDC 611658).

Conclusion

The research yielded the following results:

(i) The effects of parameters such as molar ratios of starting materials, temperature, time, and nature of solvents on the product yield of nitrobenzoic acid isomers were studied;

(ii) According to the reaction mechanisms of N,N-DEH salt formation, it was determined that the salt was formed by donating a proton to the nitrogen atom holding the ethyl groups of N,N-DEH, and the second acid molecule formed a complex by forming a strong hydrogen bond with the carbonyl oxygen of the complex;

(iii) As a result of the reaction, products were obtained with 81% yield of *o*-nitrobenzoic acid (I), 87% yield of *m*-nitrobenzoic acid (II), and 97% yield of *p*-nitrobenzoic acid (III), and their structures were analyzed using X-ray structural analysis, IR spectrum, and NMR^{1}H spectra, and the structural formulas were given.

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