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Effect of the Structure of Cyclic *N*-Nitramines on the Rate and Mechanism of Their Thermolysis

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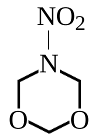
Received January 15, 2007

Abstract—Manometry together IR spectroscopy, mass spectrometry, and chromatography were used to study thermolysis of six-membered cyclic *N*-nitramines containing one, two, or three nitramine fragments, as well as oxo, *gem*-dinitro, azide, nitroimino, amino, and tetrazolyl functions. An effect of the conformation and functional groups on the rate, mechanism, and activation parameters of the limiting stage of themolysis was revealed.

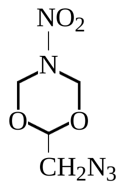
DOI: 10.1134/S1070363207070237

At present thermolysis of 1,3,5-trinitro-1,3,5-triazinane (**VIII**) has been studied in sufficient detail to conclude that the primary reaction act is homolytic cleavage of the N–NO_2 bond [1–6]. The same mechanism has been suggested for analogs of compound **VIII**, containing one or two nitramine functions [5]. However, there is no kinetic data in the literature on thermolysis of six-membered nitramines containing other functionalities.

In the present work we studied thermolysis of compounds **I**–**IX** with the aim to reveal the effect of the conformation and functional groups on the rate, mechanism, and activation parameters.



I



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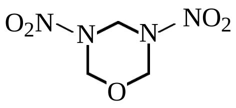

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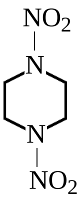

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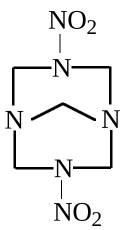

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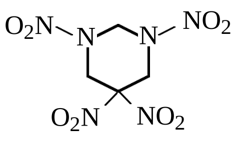

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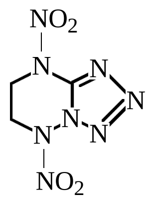
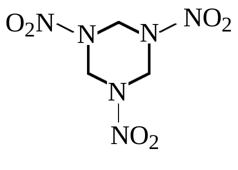
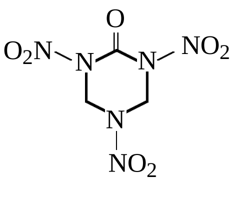


VII



VIII



IX

Thermolysis of nitramines **I**–**IX** was studied in melt and in 2 wt% solutions in inert solvents (dibutyl phthalate and dinitrobenzene). In was found that the thermolysis rate in 1–10 wt% solutions is practically the same. Thermolysis of compound **V** was studied in the solid phase.

Kinetically, thermolysis of compounds **I**–**IX** has much in common. The reaction involves acceleration and is characterized by S-shaped kinetic curves (Fig. 1). The S shape of the kinetic curves in solution is less pronounced than in melt. The initial thermolysis rate in melt depends on the sample-to-reaction

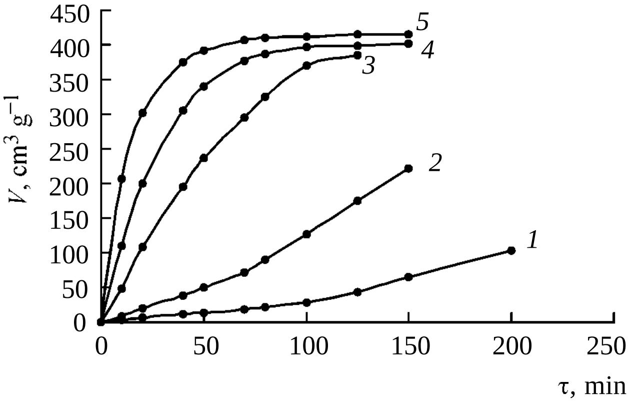


Fig. 1. Kinetic curves of the thermolysis of compound **III** in melt at m/V $(1.1–1.3) \times 10^{-3}$ g cm^{-3}. Temperature, °C: (*1*) 180, (*2*) 190, (*3*) 200, (*4*) 210, and (*5*) 220.

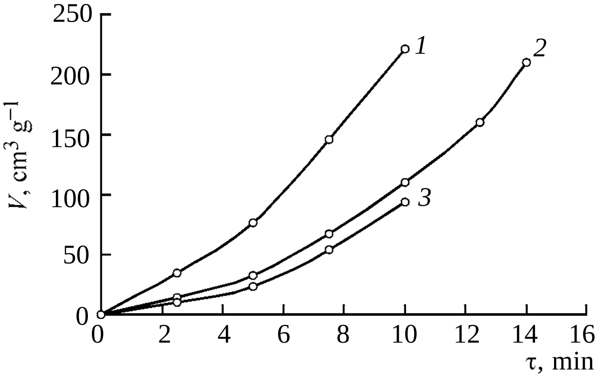


Fig. 2. Effect of m/V on the thermolysis of compound **III** in melt at 210°C. m/V, g cm^{-3}: (*1*) 5.7×10^{-4}, (*2*) 1.3×10^{-3}, and (*3*) 1.2×10^{-2}.

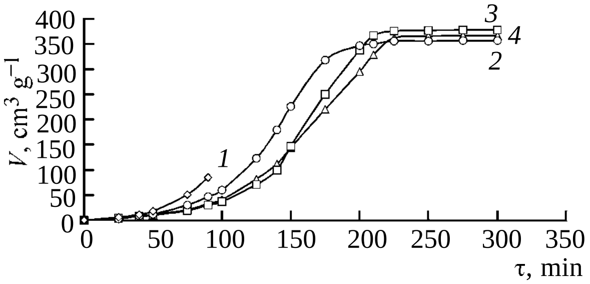


Fig. 3. Effect of m/V on the thermolysis of compound **V** in the solid phase at 160°C. m/V, g cm^{-3}: (*1*) 8.1×10^{-3}, (*2*) 1.0×10^{-3}, (*3*) 4.1×10^{-4}, and (*4*) 3.3×10^{-4} (glass capillaries packing).

volume ratio (m/V). The higher m/V, the longer inductive period and the lower thermolysis rate (Fig. 2). This effect is explained by the contribution of a gas-phase reaction to the overall reaction rate [6]. Therefore, the dependences of thermolysis rate on temperature were studied at close m/V values: $(1.1–1.3) \times 10^{-3}$ g cm^{-3}.

In the case of nitramine **V** in the solid phase, as m/V was increased 20 times, the inductive period became shorter, while the initial thermolysis rate remained almost unchanged (Fig. 3). This is explained by the fact that the reaction occurs exclusively in the condensed phase. The reactions of nitramines **I**–**IX** in different aggregative states (melt, gas, and solid) are all not complicated by heterogeneous and chain processes, as evidenced by the independence of thermolysis rate on vessel surface-to-volume ratio (S/V). The initial rate and inductive period of thermolysis of nitramine **V** are unaffected by crystal size (0.01–0.2 mm), implying that topochemistry contributes nothing into the acceleration of the solid-phase reaction. Most likely, thermolysis is accelerated due to the action of reaction products on the starting nitramine.

The principal kinetic results are given in Table 1 together with published data for 1,3-dinitro-1,3-diazinane (**X**), 2-nitro-5-nitroimino-1,3,5-triazinane (**XI**), and 5,5-dinitro-1,3-dioxane (**XII**).

As seen from Table 1, the thermolysis rates of compounds **I** and **IV** are minimal and that of compound **VII** is maximal. The thermolysis rates differ by about 7 orders of magnitude. Compounds **III**, **VIII**, and **X** have almost the same thermolysis rates in solution, even though their chemical structures are different.

It was previously suggested that the thermolysis rate of six-membered cyclic nitramines depends primarily on the conformation of the nitramino group [7]. According to neutron diffraction data [13], the nitramino groups in a crystalline compound **VIII** are nonequivalent: The amino nitrogen in two of them is pyramidal, and in the third it is planar. Such nonequivalence is associated with the crystal field effect and results in an appreciable difference in the lengths and, consequently, strengths of the N–NO$_2$ bonds. The length of the weakest N–NO$_2$ bond is 1.398 Å, and the sum of amino nitrogen bond angles is 347.9°.

According to gas-phase electron diffraction data [14], the nitramino groups in molecule **VIII** are equivalent, and their geometry is close to planar (sum of amino nitrogen bond angles 356.3°). The N–N bond length increases to 1.413 Å. In solution, there is an intermediate situation between the strong effect of intermolecular interaction in crystal and lack of such interaction in the gas phase. As judged from the dipole moments of compounds **III** and **VIII**, their nitramino groups should be equivalent in solution

Table 1. Kinetic parameters of thermolysis of cyclic *N*-nitramines

Comp. no.	Conditions[a]	Temperature range, °C	E_a, kJ mol^{-1}	log *A*	$k_{200°C}$, s^{-1}
I	Gas phase	200–230	168.7	13.95	2.1×10^{-5}
	Solution in DBP	185–220	174.6	14.46	1.5×10^{-5}
	Solution in DNB	185–225	173.9	14.40	1.6×10^{-5}
II	Solution in DBP	175–210	163.9	14.38	1.9×10^{-4}
	Solution in DNB	180–220	162.3	14.29	2.3×10^{-4}
III	Solution in DBP	170–210	169.2	14.71	1.1×10^{-4}
	Melt	180–220	171.2	14.93	1.1×10^{-4}
IV	Solution in DNB [8]	225–245	155.3	12.00	7.0×10^{-6}
	Melt [8]	216–250	198.4	17.30	2.5×10^{-5}
	Solution in TNB [9]	230–245	191.7	15.70	3.0×10^{-6}
	Melt	180–220	177.1	14.86	2.0×10^{-5}
V	Solution in DBP	150–185	148.7	13.38	9.1×10^{-4}
	Solution in DNB	150–180	153.2	13.91	9.9×10^{-4}
	Solid phase	150–180	172.9	15.64	3.5×10^{-4}
VI	Solution in DBP	145–170	157.9	14.47	1.1×10^{-3}
	Solution in DNB	150–175	160.4	14.78	1.2×10^{-3}
	Melt	165–185	159.1	14.71	1.4×10^{-3}
VII	Solution in NB	50–70	123.1	16.25	4.55×10^{2}
	Solution in DBS	50–75	128.8	16.87	4.45×10^{2}
VIII	Solution in DNB [6]	160–200	166.3	14.30	8.7×10^{-5}
	Solution in DBP	170–210	167.5	14.62	1.3×10^{-4}
	Melt	205–224	176.3	15.95	3.0×10^{-4}
IX	Solution in DBS	160–180	149.3	14.53	1.1×10^{-2}
	Solution in DNB	160–180	150.6	14.74	1.3×10^{-2}
X	Solution in EtOH [10]	200–300	167.4	14.60	1.3×10^{-4}
	Melt [10]	240–260	175.4	15.40	1.1×10^{-4}
XI	Solution in PB [11]	140–180	100.9	8.25	1.3×10^{-3}
XII	Solution in DBP [12]	195–215	174.6	14.75	3.0×10^{-5}
	Melt [12]	180–200	176.8	15.09	3.7×10^{-5}

[a] (DBP) Dibutyl phthalate, (DNB) dinitrobenzene, (TNB) trinitrobenzene, (NB) nitrobenzene, (DBS) dibutyl sebacinate, and (PB) phenyl benzoate.

[15]. As to the conformation of the amino nitrogen, is might be expected to be more pyramidal than in the gas phase. This conclusion follows from a comparison of the thermolysis rates of nitramines **III**, **IV**, **VIII**, and **X** and from the results of Burov and Nazin [7] who established that the thermal stability of nitramines is controlled by the conformation of the amino nitrogen atom. The most stable among nitramines **I**–**IX** was compound **IV**. The rate of its thermolysis was lower by 1–2 orders of magnitude compared with its isomer **X** and nitramines **III** and **VIII**, which was explained in terms of suggested planarity of both nitramino groups in compound **IV** [7]. However, no structural data for nitramine **IV** have still been available. It is presently known that compound **IV** in crystal has a chair conformation and two pyramidal nitramino groups (sum of amino nitrogen bond angles 347.0°). At the same time, the N–N bonds in compound **IV** are shorter than in **VIII** and equal 1.37 Å [16].

The suggestion of Burov and Nazin [7] that the conformation of the amino nitrogen atom plays a decisive role in the thermal stability of nitramines is ruled out by structural data. At the same time, one can try to correlate the themolysis rates of nitramines directly with the length of the weakest N–N bond.

Structural data for compound **I** are lacking. However, as judged from its thermolysis rate, the N–N bond in this compound, like in compound **IV**, is shorter than in nitramine **VIII**.

Compound **IX** contains a flattened conjugated dinitrourea fragment and has a *half-chair* (*half-boat*) conformation [17]. All the N–N bond are longer (the

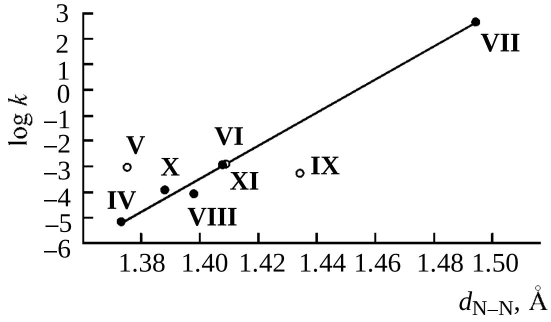


Fig. 4. Plot of the thermolysis rate constants in solutions vs. lengths of the weakest N–N bond in six-membered cyclic nitramines. The points of compounds **V, IX**, and **XI** were not used in the correlation.

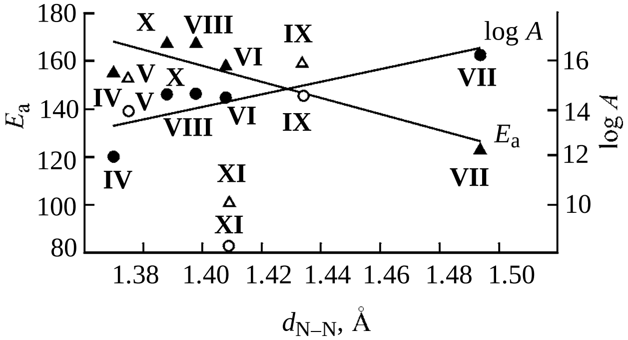


Fig. 5. Plot of the activation parameters on thermolysis in solutions vs. lengths of the weakest N–N bond in six-membered cyclic nitramines.

largest bond length is 1.434 Å) than in compound **VIII**, which explains the lower thermal stability of compound **IX**.

Unlike what is observed in **IV**, in nitramine **X** [18] one of the nitramino groups is almost planar (sum of amino nitrogen bond angles 356.5°), and the N–N bond length is 1.345 Å. The other N–NO_2 group is pyramidal (344.4°), and the N–N bond length is 1.388 Å, which is larger than in compound **IV**. As a result, nitramine **X** thermolyzes faster (Table 1).

The thermolysis rate of compound **V** in solution is about an order of magnitude higher compared with that of nitramine **VIII** and close to those of compounds **VI** and **XI**. Compound **V** in crystal has a *char–chair* conformation which is more rigid than the conformation of nitramine **VIII** [19]. The weakest N–N bond length is 1.386 A, and the sum of amino nitrogen bond angles is 346.9°. Note that the *chair–boat* and *boat–boat* structures are less favored by energy.

Compounds **II** and **VI** contain two potential reaction centers. The conformation of compound **II** is unknown. The structure of compound **VI** was studied by X-ray diffraction to show that the nitramino groups in it are nonequivalent [20]. The length of the weakest N–N bond 1.408 Å, and the sum of amino nitrogen bond angles is 349.7°.

The fastest thermolyzed is compound **VII**. Among known N–N bond lengths in nitramines, the length of one of the N–N bonds in this compound is the largest (1.494 Å) [21] (the nitramino group whose pyramidal amino nitrogen atom is directly attached to one of the tetrazole ring nitrogens). The sum of amino nitrogen bond angles is 329.0° [21], which is the lowest value among those found in the nitramines studied.

According to X-ray diffraction data, the length of the N–N bond in the secondary amino group in compound **XI** is 1.409 Å, and the sum of amino nitrogen bond angles is 337.6° [22].

We found a fair correlation between the lengths of the weakest N–N bonds in nitramines whose structural data are available with the thermolysis rate constants of these compounds in solutions [Fig. 4, regression equation (1)].

$$\log k_{200°C} = 63.708 d_{N–N} - 92.603. \qquad (1)$$

Here $d_{N–N}$ is the length of the weakest N–N bond, Å; correlation coefficient r 0.995.

The points for compounds **V** and **IX** do not fit correlation (1), which, as will be shown below, is explained in terms of a mechanism different from N–NO_2 homolysis. The point for compound **XI** fits the correlation (Fig. 4), but we consider this result accidental. Compound **XI** can thermolyze by a different mechanism (say, involving the nitroimino function [11]), as evidenced by the magnitudes of activation parameters (E_a 100.9 kJ mol^{-1}, log$A$ 8.25), uncharacteristic of N–NO_2 bond holysis.

The activation energy of the liquid-phase (melt, solution) thermolysis of compounds **I**–**X** varies from 123 to 198 kJ mol^{-1}, and the preexponential term varies from 13.28 to 16.25. With elongation of the weakest N–N bond, the activation energies of nitramines tend to decrease, and the preexponential factors tend to decrease (Fig. 5).

As mentioned above, the limiting stage of thermolysis of compound **VIII** is N–N homolysis with the following parameters: E_a 166–176 kJ mol^{-1} and log$A$ 14.30–15.95. Taking into account that the thermolysis rate in melt depends on *m/V* (Fig. 2), it is reasonable to suggest that the limiting stage has parameters

Table 2. Composition of the principal gaseous thermolysis products of nitramines **III** and **VIII** at various temperatures (mole/mole substance, $M_{rel} \times 10^2$)

Comp. no.	Temperature, °C	Conversion, %	NO	N_2O	N_2	CO	CO_2
III	180	5	0.06	0.07	0.12	0.08	0.07
	190	5	0.27	0.28	0.54	0.28	0.16
	190	20	1.61	2.95	3.88	2.49	1.87
	190	30	6.14	9.52	9.03	8.06	3.68
	210	98	1.78	3.73	10.00	5.55	7.81
VIII	210	98	2.66	6.31	13.2	4.21	4.98

Table 3. Composition of uncondensed gases in solid-phase thermolysis of compound **V** (mol%)

Temperature, °C	Conversion, %	Composition of thermolysis products			
		N_2	N_2O	CO	CO_2
160	10	0.98	96.19	0.90	1.93
	20	0.69	96.47	0.83	2.01
	50	0.64	95.57	1.09	2.69
	75	0.66	93.94	1.29	4.11
	100	0.64	91.87	2.39	5.10
150	10	0.68	96.32	0.83	2.18
	20	0.46	96.55	0.76	2.23
170	100	0.95	88.22	2.87	7.94

characteristic of solution: E_a 166.3–167.5 kJ mol^{-1} and log*A* 14.30–14.62.

Homolysis by the N–N bond as the limiting thermolysis stage is also realized with nitramines **I**, **III**, **IV**, and **X**, as judged from the activation parameters and dependences in Figs. 4 and 5. In the thermolysis of compound **VII** in the solid phase, as the primary product we found nitrogen dioxide, which allowed us to suggest N–N homolysis as the initial stage [21]. Presumably, in solution, too, this mechanism is operative.

Qualitatively, gaseous products of thermolysis of compounds **I**, **III**, **VIII**, and **IX** are almost the same: N_2, N_2O, NO, CH_2O, CO, CO_2, H_2O, and HCN (traces). The quantities of the nitrogenous gaseous products N_2, N_2O, and NO, as well as CO and CO_2 for compound **III** in melt are listed in Table 2.

Nitrogen dioxide formed in the primary act was not found among gaseous products of thermolysis of compound **III**, since this product was rapidly consumed in redox reactions [5]. Analysis of the thermolysis products of compound **II** in melt showed that with increasing conversion the intensity of the absorption band of the azido group in the IR spectra decreases (2140 cm^{-1}). At the same time, the intensity of the N–NO_2 bands (1555, 1295 cm^{-1}) remains virtually invariable. At a 1–3% conversion we found no other products as molecular nitrogen and 2-methyleneimino-5-nitro-1,3,5-dioxazinane. Nitrogen dioxide appears at deeper conversions (7–12%), when the composition of thermolysis products becomes fairly complex. These data provide evidence to show that thermolysis of compound **II** is limited by decomposition of the azido group.

The reaction center can also be expected to change in the case of compound **VI**. As seen from Table 1, the dinitromethylene group in the ring of compound **XII** is close in stability of the nitramine group of compound **I**. In going from compound **X** to compound **VIII**, the thermolysis rate changes only slightly. This can be associated with the steric effect of the dinitromethylene groups on one of the nitramine groups [20]. Similar effect was observed on replacement of one methyl group in *N*,*N*-dimethyl-*N*-nitramine by a 2,2-dinitropropyl group [23]. Thus, we can conclude that thermolysis of compound **VI**, like with compound **VIII**, involves initial homolytic cleavage of the N–N bond.

The results of analysis of gaseous products of thermolysis of compound **V** at various temperatures (Table 3) show that the principal component is N_2O, whereas NO is lacking. The N_2/N_2O ratio is lower by about two orders of magnitude than with compounds **III** and **VIII**. With this in mind, we can suggest that thermolysis of compound **V**, rather than initiating with N–N homolysis, involves intra- or intermolecular transfer of the nitro oxygen onto the methylene carbon followed by C–N cleavage to form CH_2O and N_2O [24]. The presence of CH_2O among the thermolysis products of nitramine **V** was proved by mass spectrometry and chemical analysis (dimedone test). This mechanism may well be realized, since, first, because of the presence in the crystal structure of compound **V** of many shortened contacts (in particular, the distances between oxygen and methylene carbons are 2.62–2.69 Å [19], which is smaller that the sum of the van der Waals radii of these atoms [25]), and, second, because of the absence from the reaction products of NO and presence of minor amounts of CO and CO_2 (Table 3) which inevitably present in moderate amounts in redox reactions involving NO_2. However, as noted by Brill [26], N_2O and NO_2 appear together in thermolysis of compound **V** slightly above the decomposition point, but N_2O always prevails near the decomposition point.

Furthermore, compound **V** contains two tertiary amino functions and can exert base catalysis whose details were well known [27]. As a result, nitramide can form and decompose to generate N_2O and H_2O.

The principal thermolysis products of compound **IX** are CH_2O, N_2O, and CO_2, while NO_2, NO, and CO were found in minor amount, which allowed us to suggest that the limiting stage involves reaction between the nitramide and carbonyl groups via a four-membered cyclic transition state [28]. This is an explanation for the fact that the point for compound **IX** falls out from the correlation dependence (Fig. 4).

As already noted, thermolysis of compound **XI** is limited by decomposition of the nitroimino group [11]. Evidence for this assumption is provided by the activation parameters and by the high content of N_2O in primary thermolysis products.

In summary we can note that a combination of secondary nitramino groups with oxo, *gem*-dinitro, and tetrazolyl group in a six-membered hetecycle does not change the limiting stage of thermolysis, viz. N–N homolysis, but can increase thermolysis rate by 2–7 orders of magnitude. In the presence of the azido, tertiary amino, carbonyl, or nitroguanidyl group, the limiting thermolysis stage can be different. Conformational features of the nitramino group and six-membered heterocycle affect the thermal stability of the nitramine through the length of the weakest N–N bond. A good correlation was found between thermolysis rate constant and the weakest N–N bond length in nitramines.

EXPERIMENTAL

Gaseous thermolysis products were analyzed on an LKhM-80 chromatograph (column packed with Polisorb-1, temperature 18–20°C), a UR-20 IR spectrometer, as well as MX-1312 (column with squalane) and MI-12-01T instruments (pressure 10^{-5}–10^{-7} mm Hg, ionization energy 70 eV, accelerating voltage 5 kV).

Compounds **I**–**IX** were purified by recrystallizati on from appropriate solvents and were chromatographically pure (99.6–99.8%). Thermolysis kinetics were studied under isothermal conditions by the manometric technique using a Bourdon manometer [29] at the residual pressure in the reaction vessel of 10^{-2}–10^{-3} mm Hg.

The rate constants were calculated from the initial rates with an error of no more than 10%. The errors in the activation energy and log *A* were no more than 6.5 kJ mol^{-1} and 0.6 log units, respectively.

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