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> PHYSICOCHEMICAL STUDIES OF SYSTEMS AND PROCESSES

New Approaches to the Development of High-Performance Hydrocarbon Propellants

M. V. Savos'kin, L. M. Kapkan, G. E. Vaiman, A. N. Vdovichenko, O. A. Gorkunenko, A. P. Yaroshenko, A. F. Popov, A. N. Mashchenko, V. A. Tkachev, M. L. Voloshin, and Yu. F. Potapov

Litvinenko Institute of Physical Organic and Coal Fuel Chemistry, National Academy of Sciences of Ukraine, Donetsk, Ukraine

Yangel' Yuzhnoe State Design Office, National Space Agency of Ukraine, Dnepropetrovsk, Ukraine

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Abstract—An approach to express screening of promising hydrocarbon propellants, based on the calculation of the specific impulse of an engine from the results of quantum-chemical calculation of their heats of combustion, was suggested. The approach ensures high accuracy irrespective of the composition and structure of the compounds under consideration. Polycyclic and framework hydrocarbons with small rings were suggested as promising propellants.

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The development of new hydrocarbon propellants for environmentally clean rockets is a priority research field, because the use of energetic propellants increases the pay load of carrier rockets and makes their launch more cost-efficient.

One of approaches to enhancing the performance of hydrocarbon propellants is increasing their density by using cyclic and framework compounds [1–7]. Such propellants are usually prepared by hydrogenation of dicyclopentadiene followed by catalytic rearrangement of the product from the *endo* to *exo* form, which is required for decreasing the melting point. Such propellants as, e.g., JP-10, RJ-4, and RJ-41 with a density of 0.94 g cm⁻³ were prepared by this procedure. With norbornane taken as starting compound, a propellant (RJ-5) with a still higher density, 1.08 g cm⁻³, was prepared. Another way to increase the density is the condensation of dicyclopentadiene with adamantane derivatives; however, this leads to a significant increase in the melting point.

To enhance the performance of hydrocarbon propellants, their heat of combustion is increased, in particular, by using structurally strained hydrocarbons containing three- and four-membered rings [8]. These compounds contain fragments with strongly distorted C–C bond angles, which leads to appreciably larger heats of formation and hence higher heats of combustion. The suggested propellants based on 1-methyl-1,2-dicyclopropylcyclopropane [9] and dicyclobutyl [10-13] are characterized by record-breaking heats of combustion.

We believe that the use of polycyclic and framework hydrocarbons with small rings as propellants can further enhance the energy characteristics of rockets owing to high density and increased heat of combustion of such propellants.

Studies on synthesis and determination of the characteristics of new fuels require considerable expenses; therefore, it is appropriate to start searching for compounds having the required properties with the calculation of the relevant physicochemical characteristics. With the modern computers, the existing quantumchemical methods allow quick screening of the potentially promising compounds with incommensurably lower time and money expenses compared to experimental studies. This approach also allows calculation of the characteristics of difficultly available and even hypothetical substances.

Various empirical additive schemes for calculating the heat of combustion require the knowledge of such molecular parameters as the bond energies or force constants. Additive schemes give particularly inaccurate results when applied to compounds of different types, e.g., in going from normal hydrocarbons to cyclic hydrocarbons, especially those with small rings. Therefore, such calculations do not ensure the level of versatility and accuracy required for comparing the parameters of compounds with diverse chemical structures. Molecular mechanics methods suffer from the same drawback.

Since the heat of combustion of the compounds of interest varies in a relatively narrow range (10% at maximum and 2-3% in most cases), the only method giving the reliable and comparable data is direct ab initio calculation of the heat of combustion.

The overall reaction equation for the combustion of hydrocarbon propellants in oxygen at a stoichiometric component ratio,

$$C_n H_m + (n + m/4)O_2 = m/2H_2O + nCO_2,$$
 (1)

corresponds to the real calorimetric experiment. Although the error in calculating the heat of combustion, as will be shown below, is 4-8 kJ mol⁻¹ and somewhat exceeds the error of experimental determinations, the calculation accuracy attained is quite sufficient for comparing the power intensity of various hydrocarbons.

The calculations reported in this paper were performed ab initio using the GAMESS program package [14] which allows, in particular, calculation of the total energies of molecules and of the correlation and thermodynamic corrections to them. The molecular geometries were fully optimized within the framework of the restricted Hartree-Fock (RHF) method in the TZV(d,p) basis set [15] with the polarization d functions on the C atoms and p functions on the H atoms. To determine the thermochemical correction at 25°C, the harmonic vibration problem [16] was solved in the same approximation. For the molecular geometry corresponding to the total energy minimum in the TZV(d,p) basis set, the electron correlation was taken into account according to the Møller-Plesset perturbation theory (MP2) [17, 18] in a wider basis set, TZV(2df,2p). Several test calculations of small molecules showed that the geometry optimization in this basis set did not lead to appreciable decrease in the total energy. The oxygen molecule in the ground triplet state was calculated by the restricted open-shell Hartree-Fock (ROHF) method, and the MP2 electron correlation correction for this molecule was determined according to [19]. The required total energies with the MP2 correction, calculated in the TZV(2df,p)basis set (in atomic energy units) are as follows: H₂O -76.3204, CO₂ -188.3156, and O₂ -150.1218. The

thermochemical corrections to their enthalpies at 25° C are 70.43, 42.80, and 20.64 kJ mol⁻¹, respectively.

The calculated heats of combustion in oxygen of saturated hydrocarbons of different chemical structures and several unsaturated hydrocarbons in the gas phase are listed in the table.

To evaluate the accuracy of calculating the heat of combustion within the framework of the abovedescribed scheme, we calculated the total energies of aliphatic hydrocarbons for which these quantities are well-known. As follows from the table, the relative errors of the calculated heats of combustion of the homologous series of hydrocarbons (from methane to octane) are within 0.12-0.37%. Appreciably higher accuracy is attained for molecules containing small rings. In this case, the error does not exceed 0.1% and in most cases is within 0.03-0.04%. The experimental heats of combustion presented in the table refer to the gas phase.

Note that, when comparing the power intensity of various compounds, the heat of combustion is usually referred to 1 g of the combustible but not of the fuel (combustible + oxidant), which can lead to wrong conclusions when choosing the most efficient fuel. Indeed, assume that combustion of fuel of mass m results in evolution of Q J of heat. Then, neglecting the thermal dissociation of combustion products, we can determine the velocity of their outflow v from the law of energy preservation:

$$mv^2/2 = \eta Q, \qquad (2)$$

where η is the engine efficiency factor.

Hence,

$$v = (2\eta H)^{1/2}$$
(3)

(Glushko's formula), where

$$H = Q/m \tag{4}$$

is the heat of combustion per unit mass of the fuel $(J \text{ kg}^{-1})$, which, as seen from formula (3), is the major factor determining the outflow rate and hence the engine thrust.

Let us illustrate these reasonings by a simple example. Judging from the specific heat of combustion per kilogram of combustible h, methane is the most efficient (see table); however, its H value is relatively low, determining its moderate power characteristics as propellant. In the combustion of methane,

$$CH_4 + 2O_2 = CO_2 + 2H_2O,$$
 (5)

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Com- pound	Hydrocarbon	Formula	Heat of combustion, kJ mol ⁻¹		Specific heat of combustion, kJ kg ⁻¹		Specific im- pulses,**
no.			caculation	experiment*	of combustible, h	of fuel, H	s
1	Methane	CH ₄	797	794 [20]	49657	9953	339.1
2	Ethane	C_2H_6	1423	1419 [21]	47 312	10014	340.2
3	Propane	C_3H_8	2037	2040 [21]	46 184	9978	339.6
4	Butane	C_4H_{10}	2650	2658 [22]	45 595	9959	339.2
5	Octane	C_8H_{18}	5106	5112 [20]	44 696	9929	338.7
6	Dicyclobutyl	\leftrightarrow	4911	4909	44 561	10269	344.5
7	<i>exo</i> -Tetrahydrodicyc- lopentadiene (IP-10)	\bigcirc	5780	5770 [5]	42 421	9893	338.1
8	Tetracyclo- $[3.3.1.0^{2,4}0^{6,8}]$ nonane	\Diamond	5195	5193 [23]	43 225	10305	345.1
9	ω-Pinene	\rightarrow	5893	5891 [24]	43 253	10086	341.4
10	β-Pinene	=⊗	5905	5899 [24]	43 343	10108	341.8
11	Pinane	\neg	6024		43 571	10 003	340.0
12	Tricyclo[5.2.0.0 ^{2,5}]-		5347		43 754	10 2 4 0	344.0
13	Tricyclo[5.2.0.0 ^{2,5}]-		5332		43 631	10211	343.5
14	Tricyclo[5.2.0.0 ^{2,5}]-		5544		45 366	10617	350.3
15	Tricyclo[4.1.0.0 ^{2,4}]-	\bigcirc	4113	4113 [25]	43 682	10330	345.5
16	Tricyclo[4.1.0.0 ^{2,4}]-		4139		43 957	10395	346.6
17	Tricyclo[4.1.0.0 ^{2,4}]-	\bigtriangledown	4623		49 096	11610	366.3
18	Tricyclo[3.2.0.0 ^{2,4}]-		4199		44 595	10546	349.1
19	Tricyclo[3.2.0.0 ^{2,4}]- heptane II		4239		45 020	10647	350.7
20	4,7,7-Trimethyltricyc- lo[4.1.1.0 ^{2,4}]octane****	$\neq \downarrow$	6558		43 644	10148	342.4
21	1,4,4-Trimethyltricyc- lo[5.1.1.0 ^{3,5}]octane****	M	6556		43 629	10145	342.4
22	2,6-Dimethyl2ricyclo- [3.1.1.0 ^{3,6}]heptane	\rightarrow	5384		44 057	10311	345.2
23	Tricyclo[3.3.1.0]nonane	$\langle \rangle$	5239		42 870	10033	340.5
24	Tricyclo[3.3.2.0]decane	$\overline{\square}$	5834		42 825	9987	339.7

Calculated heats of combustion of hydrocarbons and specific engine impulses

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Table.	(Contd.)
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Com- pound	Hydrocarbon	Formula	Heat of combustion, kJ mol ⁻¹		Specific heat of combustion, kJ kg ⁻¹		Specific im- pulses,**
no.			caculation	experiment*	of combustible, h	of fuel, H	s
25	Bicyclo[1.1.0]butane	\Diamond	2520		46 592	10954	355.8
26	Tricyclo[2.1.0.0 ^{2,5}]- pentane	\triangleleft	3087		46 699	11 262	360.7
27	Bicyclo[2.1.0]pentane	\square	3089		45 342	10574	349.5
28	Tricyclo[3.1.0.0 ^{2,4}]hex- ane	\bigcirc	3595		44 864	10696	351.6
29	Prismane	⋬	3646		46 678	11 462	363.9
30	Tricyclo[3.1.0.0 ^{2,6}]hex-	\bigtriangledown	3550		44 301	10561	349.3
31	ane Quadricyclane	\bigcirc	4050		43 956	10655	350.9
32	Cubane	Ø	4733		45 439	11 158	363.9
33	Spiropentane	\checkmark	3115		45731	10664	351.0
34	Norbornane	A	4139		43 040	9947	339.0
35	Norbornene	A	4043		42936	10154	342.5
36	Norbornadiene	A	3968		43 065	10439	347.3
37	Nortricyclene	4	4023		42 731	10105	341.7
38	Adamantane	Ð	5720	L	41 986	9791	336.4

* For the gas phase.

** Calculation by Glushko's formula (8) at $\eta = 0.556$.

*** Bold dots denote hydrogen atoms located over the molecular plane.

**** Because of the large molecular size, the calculations were performed in a somewhat less accurate basis set TZV(df,2p), which leads to ~1-s underestimation of the specific impulse.

the oxygen consumption is 3.9891 mass units per mass uinit of methane. Therefore, for complete combustion of methane in oxygen, we have

$$H = h/(1 + 3.9891) = 49657/4.9891 = 9953 \text{ kJ kg}^{-1}.$$

Similarly, in combustion of, e.g., dicyclobutane,

$$C_8H_{14} + 11.5O_2 = 8CO_2 + 7H_2O$$
 (6)

the oxygen consumption is as low as 3.339 mass units per mass unit of the combustible; hence,

$$H \approx h/(1 + 3.339) = 44561/4.339 = 10269 \text{ kJ kg}^{-1}.$$

Thus, although the heat of combustion per kilogram of combustible (*h*) of methane, 49.657 kJ kg⁻¹, appreciably exceeds that of dicyclobutyl, 44.561 kJ kg⁻¹, methane is less efficient because of the lower heat of

combustion per kilogram of fuel (*H*): 9953 kJ kg⁻¹ against 10269 kJ kg⁻¹ for dicyclobutyl. Note that the reasonings given here and below refer exclusively to the stoichiometric ratio of the fuel components, combustible and oxidant. Actually, to attain the maximal performance of a specific type of propellant, the optimal amounts of the oxidant are determined experimentally, and they are usually appreciably lower than the stoichiometric amount. In this case, our approach to assessing the performance of propellants can be used as a preliminary estimate.

To convert the specific heat of combustion of a hydrocarbon h to the specific heat of combustion of a fuel H, it is convenient to use a formula following from Eq. (1):

$$H = h(11.91 + x)/(43.66 + 8.936x), \tag{7}$$

where x = m/n is the H/C atomic ratio in the given hydrocarbon $C_n H_m$.

An important conclusion follows from Eq. (7): With a decrease in the H/C ratio x in a hydrocarbon, the conversion factor from h to H increases. Thus, a propellant with a low hydrogen content can exhibit a higher specific impulse, even despite lower power intensity h per kilogram of the hydrocarbon.

By multiplying the outflow velocity of gaseous products v from formula (3) by the fuel consumption rate (kg s⁻¹), we obtain the tractive force in newtons. In the literature on rocket engines, the specific impulse p_{sp} , measured in kilogram-force (kgf) units and equal to the tractive force at a fuel consumption rate of 1 kg s⁻¹, is commonly used. Conversion of formula (3) to p_{sp} gives

$$p_{\rm sp} = (2\eta H)^{1/2}/g,$$
 (8)

i.e., the dimension of p_{sp} is kgf kg⁻¹ s or simply s after the "cancellation" of kgf and kg, which is a common practice. The results of calculation by formula (8) are given in the table. The efficiency factor η was taken equal to 0.556 to bring the calculated and experimental [10–12] specific impulses of dicyclobutyl to coincidence; this value of η was also applied to calculate the specific impulses of all the other compounds listed in the table. Thus, the parameter η plays the role of a calibration factor taking into account, to a first approximation, the nonstoichiometric ratio of the combustible and oxidant, partial dissociation of combustion products, etc. It is assumed that these factors affect the specific impulse of the examined substances to approximately the same extent as that of dicyclobutyl.

The specific impulses given in the table range from 336.4 for adamantane to 366.3 for stereoisomer III of tricyclo[4.1.0.0^{2,4}]heptane (no. 17). Note that the specific impulses of adamantane and JP-10 propellant are close to that of kerosene, 337.9 s [10–12]. The natural terpenes α - and β -pinene present in turpentine exhibit higher specific impulse, 341.4–341.8 s. Still higher values, 345–350 s, are attained, e.g., with 2,6-dimeth-yltricyclo[3.1.1.0^{3,6}]heptane (no. 22), norbornadiene (no. 36), tricyclo[3.1.0.0^{2,6}]hexane (no. 30), and bicyclo[2.1.0]pentane (no. 27). Note that tetracyclo[3.3.1.-0^{2,4}0^{6,8}]nonane (no. 8) suggested in [23] as a promising aviation fuel, according to our calculations, has a specific impulse of 345.1 s, only slightly exceeding that of dicyclobutyl (344.5 s).

The highest values of the specific impulse (exceeding 350 s) are characteristic of such strained framework hydrocarbons as quadricyclane (no. 31), spiropentane (no. 33), bicyclobutane (no. 25), cubane (no. 32), tricyclo[$2.1.0.0^{2,5}$]pentane (no. 26), and prismane (no. 29). Many of these compounds are hypothetical and have not been synthesized up to now. The synthesis of the other compounds of this group is extremely difficult and labor-consuming, and it can hardly be realized on the commercial scale in the near future. Furthermore, such compounds have high molecular symmetry and hence should have high melting point at relatively low boiling point, which is hardly acceptable for propellants.

The three series of stereoisomers of tricyclo- $[3.2.0.0^{2,4}]$ heptane (nos. 18, 19), tricyclo $[5.2.0.0^{2,5}]$ -nonane (nos. 12–14), and tricyclo $[4.1.0.0^{2,4}]$ heptane (nos. 15–17) deserve particular consideration. As seen from the table, in going from the "transoid" chair-like to "cisoid" boat-like molecular configuration, the specific impulse increases by only 0.5–1.6 s. However, a striking result was obtained with the "skewed" isomers: the specific impulse increased by 6.8 s for tricyclo $[5.2.0.0^{2,5}]$ nonane III (no. 14) and even by 20.8 s fir tricyclo $[4.1.0.0^{2,4}]$ heptane III (no. 17) (relative to the coresponding transoid structures). Compound no. 17 even exceeds prismane (no. 29) in the specific impulse, being a "champion" among the structures chosen.

Analysis of data in the table, taking into account the set of requirements to propellants (melting and boiling points, density, viscosity, stability, etc.) shows that the most promising for further experimental studies are terpene derivatives (nos. 20, 21) and tricycloheptanes (nos. 15–17), the more so as the raw materials for their synthesis (turpnetine oil and cyclopentadiene) are cheap and readily available.

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For primary estimation of properties of potentially promising propellants, we synthesized laboratory samples of some of them. Tricyclo[$4.1.0.0^{2,4}$]heptane was prepared by the Simmons–Smith reaction [26] used for obtaining three-membered hydrocarbon rings. Cyclopentadiene prepared by thermal depolymerization of dicyclopentadiene was treated with methylene iodide in the presence of silver-plated zinc dust; in the process, it was smoothly converted to tricyclo-[$4.1.0.0^{2,4}$]heptane:



The product was identified by ¹H NMR spectroscopy as transoid stereoisomer I of tricyclo[$4.1.0.0^{2,4}$]heptane (no. 15); its calculated specific impulse is 345.5 s. The compound synthesized has a density of 0.908 g cm⁻³ (0.8912 according to [25]); its freezing point is below -103°C, and the boiling point is 104°C (103°C [25]). Unfortunately, the other stereoisomers (nos. 16, 17) were not detected in the product.

Apparently, the purposeful synthesis of highly strained stereoisomers like nos. 14 and 17 requires a higher level of preparative organic chemistry. Such a synthesis may involve the use of coordinating matrices or catalysts ensuring formation and preservation of the required strained steric configuration.

For chemical modification of natural terpenes, it appeared feasible to use a two-step synthesis scheme involving addition of dichlorocarbene to the double bond of the substrate under phase-transfer conditions (with triethylbutylammonium chloride, TEBAC, as phase-transfer catalyst), followed by reductive dehalogenation of the intermediate product with sodium in liquid ammonia. This scheme is preferable over the Simmons–Smith reaction because of the availability of the reagents and solvents and high yield and purity of the products.

With α -pinene used as precursor, this scheme leads to 4,7,7-trimethyltricyclo[4.1.1.0^{2,4}]octane (no. 20) whose calculated specific impulse is 342.4 s:



and from carene, 1,4,4-trimethyltricyclo[5.1.0.0^{3,5}]octane (no. 21) having the same specific impulse is obtained:



Since the above-described two-step synthesis with individual turpentine components occurs very smoothly and with high product yields, we made an attempt to use as starting reagent turpentine oil, a multicomponent mixture of natural terpenes. By so doing, we obtained a propellant with a density of 0.882 g cm⁻³, freezing point below -90° C, and boiling onset point of 186°C. The expected specific impulse should be 342–343 s.

CONCLUSIONS

(1) Evaluation of the specific impulse of a rocket engine, based on quantum-chemical calculation of the heat of combustion of hydrocarbon propellants, is an efficient approach to their express screening. This approach ensures high accuracy when applied to hydrocarbons of diverse compositions and structures.

(2) The use of polycyclic and framework hydrocarbons with small rings as propellants can ensure further enhancement of the power characteristics of rocket engines due to high densities and high heats of combustion of such compounds.

(3) The power characteristics of strained polycyclic hydrocarbons essentially depend on their steric configuration. For example, the stereoisomers of tricyclo[$4.1.0.0^{2,4}$]heptane differ in the specific impulse by more than 20 s. (4) Strained polycyclic and framework hydrocarbons selected according to the calculation results were synthesized by addition of carbenes to double bonds of natural terpenes and cyclopentadiene. The products obtained exhibit increased specific umpulse and density; they also meet other requirements to propellants.

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