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1 October 1978 - 30 September 1979

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THERMODYNAMICS OF ORGANIC COMPOUNDS

Bartlesville Energy Technology Center
Department of Energy
Bartlesville, Oklahoma

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Measurements were completed of the enthalpy of combustion of the ramjet fuel RJ-6. Measurements of the enthalpy of combustion of hexacyclic <i>exo,exo</i> -dihydrodinorbornadiene, hexacyclic <i>endo,endo</i> -dihydrodinorbornadiene and <i>exo</i> -tetrahydrodicyclopentadiene are in progress. Vapor pressure measurements were made on <i>exo</i> -tetrahydrodicyclopentadiene. Synthesis and purification of alkylindans and alkylnaphthalenes with high steric interactions continue at Oklahoma State University. <i>Unclassified</i>			

FINAL TECHNICAL SUMMARY REPORT

THERMODYNAMICS OF ORGANIC COMPOUNDS

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Bartlesville Energy Technology Center
Department of Energy
Bartlesville, Oklahoma

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* Synthesis and purification of research samples were provided by Professor E. J. Eisenbraun, Oklahoma State University. Samples were produced by purchase agreement for this project.

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FOREWORD

This research program consists of an integrated and inter-related effort of basic and applied research in chemical thermodynamics and thermochemistry. Knowledge of variation of physical and thermodynamic properties with molecular structure is used to select compounds for study that because of high ring strain or unusual steric effects may have good energy characteristics per unit volume or per unit mass and thus be useful in the synthesis of high energy fuels. These materials are synthesized, and their thermodynamic properties are evaluated. In cooperation with researchers at Wright-Patterson Air Force Base, ramjet fuels currently in use are subjected to careful thermodynamic evaluation by measurements of heat capacity, enthalpy of combustion and vapor pressure.

ABSTRACT

Basic and applied research have continued on the thermodynamic properties of currently used high density/high energy fuels and of pure chemical compounds that may be constituents of high energy fuels of the future.

Enthalpy of combustion was measured for one ramjet fuel currently in use, and similar measurements are in progress on three others.

In cooperation with researchers at Wright-Patterson Air Force Base, measurements were made of the vapor pressure of JP-10, *exo*-tetrahydrodicyclopentadiene, in order to derive knowledge of its possible concentration and/or toxicity in confined storage.

Synthesis and purification of a group of alkylindans and alkyl-naphthalenes continued at Oklahoma State University. These compounds are representatives of a type that will have higher combustion energies because of steric interaction of closely adjacent alkyl groups.

Results of the research were reported both orally and in AFOSR special reports and journal articles.

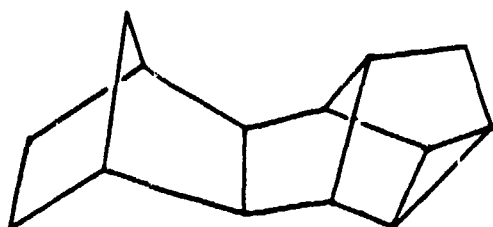
RESEARCH PROGRESS

1. NOMENCLATURE

This annual report will describe research on the thermodynamic properties of several fuels and compounds of considerable molecular complexity. In an effort to facilitate understanding, the nomenclature and molecular structure of these materials follow in Table 1.

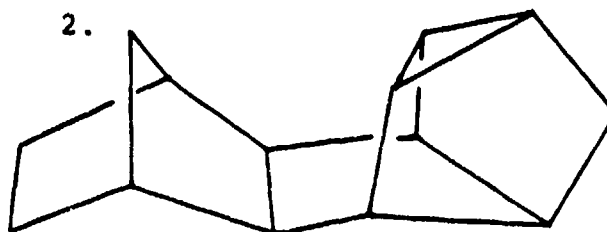
TABLE 1. Nomenclature of Materials

1.



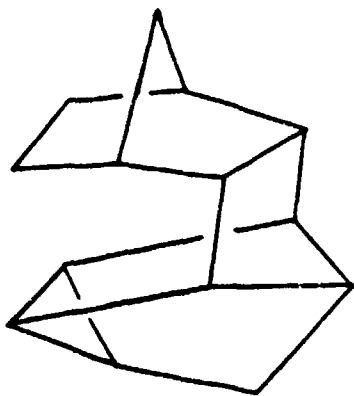
Hexacyclic
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dinorbornadiene

2.



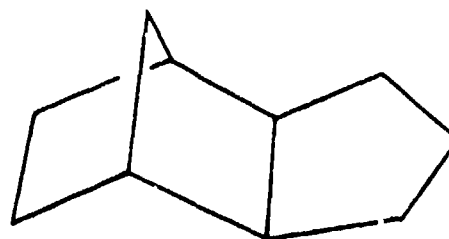
Hexacyclic
exo,endo-dihydro-
dinorbornadiene

3.



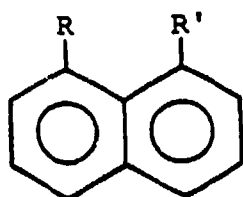
Hexacyclic
endo,endo-dihydro-
dinorbornadiene

4.



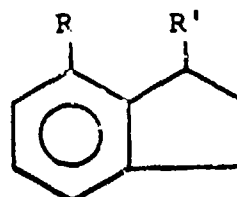
exo-Tetrahydro-
dicyclopentadiene

5.



1,8-Dialkylnaphthalenes

6.



1,7-Dialkylindans

2. ENTHALPY OF COMBUSTION OF RJ-6

Material and Techniques

RJ-6 is a blend of *exo*-tetrahydrodicyclopentadiene (compound 4 of Table 1) and the hydrogenated dimers of norbornadiene (compounds 1, 2 and 3 of Table 1). The material was supplied by Herbert T. Lander, Jr., Fuels and Lubrication Division, Air Force Aero Propulsion Laboratory (AFSC) Wright-Patterson Air Force Base, Ohio. It was used as received without further drying. Fragile flexible ampoules^{1,2} of borosilicate glass confined the samples of RJ-6; auxiliary oil (laboratory designation TKL 66) was used to initiate the combustion. Rotating-bomb calorimeter BMR II³ and platinum-lined bomb PT-3b⁴ were used without bomb rotation. For each experiment, 1 cm³ of water was added to the bomb, and the bomb was flushed and charged to 30 atm with pure oxygen; nitric acid formation during the combustion was negligible. Each experiment was started at 296.15 K, and because the masses of combustibles were properly chosen, the final temperatures were very nearly 298.15 K. Temperatures were measured by quartz crystal thermometry; the quartz thermometer was calibrated with a platinum resistance thermometer. A programmable desktop calculator was used to control the combustion experiments and record the results. Readings were taken at 100-second intervals throughout the experiment; integration of the time-temperature curve is inherent in the quartz thermometer reading.

The experimental results are based on 1961 atomic weights.⁵ For reducing weights in air to masses, converting the energy of the actual bomb process to that of the isothermal bomb process, and reducing to standard states,⁶ the following values were used for the properties of RJ-6: density, 1.01 g cm⁻³;⁷ specific heat capacity, (0.3) cal K⁻¹ g⁻¹; and $(\partial E/\partial P)_T$, (-0.003) cal atm⁻¹ g⁻¹; values in parentheses are estimates.

-
- ¹ W. D. Good and N. K. Smith, *J. Chem. Eng. Data*, **14**, 102 (1969).
 - ² G. B. Guthrie, D. W. Scott, W. N. Hubbard, C. Katz, J. P. McCullough, M. E. Gross, K. D. Williamson and G. Waddington, *J. Am. Chem. Soc.*, **74**, 4662 (1952).
 - ³ W. D. Good, D. W. Scott and G. Waddington, *J. Phys. Chem.*, **60**, 1080 (1956).
 - ⁴ W. D. Good, D. R. Donslin, D. W. Scott, A. George, J. L. Lacina, J. P. Dawson and G. Waddington, *J. Phys. Chem.*, **63**, 1133 (1959).
 - ⁵ A. E. Cameron and E. Wichers, *J. Am. Chem. Soc.*, **84**, 4175 (1962).
 - ⁶ W. N. Hubbard, D. W. Scott and G. Waddington. In *Experimental Thermochemistry*, Chap. 5. F. D. Rossini, editor. Interscience: New York. 1956. pp. 75-128.
 - ⁷ Private communication, James R. McCoy.

National Bureau of Standards sample 39i benzoic acid was used for calibration; the result from eight experiments interspersed with the RJ-6 experiments was $\epsilon(\text{calor}) = (4005.52 \pm 0.05) \text{ cal deg}^{-1}$ (mean and standard deviation of the mean).

RESULTS

The results are summarized in Table 2. The values of $\Delta E^\circ/m$ refer to the reaction of unit mass of sample. Carbon dioxide was recovered from all of the experiments. The ratio of carbon dioxide recovered to mass of sample burned was 3.2725 ± 0.0002 (mean and standard deviation of the mean). The empirical formula of RJ-6 calculated from this ratio and the assumption that only carbon and hydrogen are present is $\text{CH}_{1.4259}$.

The enthalpy of combustion given in Table 2 is the "gross" heat of combustion for which the reaction products are gaseous carbon dioxide and liquid water. For combustion to gaseous carbon dioxide and gaseous water, the "net" heat of combustion of RJ-6 is $-(17971.8 \pm 0.4) \text{ Btu/lb}$.

3. HEAT CAPACITY OF RJ-6

The heat capacity of RJ-6 was measured by differential scanning calorimetry in the range 260 to 340 K. The following linear expression was selected to fit the data by means of a least squares fit:

$$C_s = 0.0852880 + 0.0008948 T \text{ cal g}^{-1} \text{ deg}^{-1}.$$

Root-mean-square deviation of results was $0.0055 \text{ cal g}^{-1} \text{ deg}^{-1}$.

4. VAPOR PRESSURE OF JP-10

The vapor pressure of JP-10 was investigated by inclined-piston-gauge manometry. The initial expectation was that the sample supplied was substantially pure *exo*-tetrahydro-dicyclopentadiene, and that the measurements could be done as for a pure chemical compound. That expectation was not realized.

As the measurements are conducted, before each data point is obtained a small amount of the sample is pumped off to flush out traces of permanent gases that could come from slow out-gassing of components of the system. For a single-component sample, the pumping does not change the composition nor the vapor pressure. However, for a mixture of a major component with impurities of different volatility, the pumping tends to deplete the sample of more volatile impurities and concentrate the less volatile impurities, with an attendant decrease of the vapor pressure.

TABLE 2. Summary of Calorimetric Experiments with RJ-6 at 298.15 K^a
(cal_{th} = 4.184 J)

	1	2	3	4	5	6	7
m' (compound)/g	0.672225	0.688259	0.689959	0.691482	0.706798	0.713814	0.720016
m'' (auxiliary oil)/g	0.079163	0.068885	0.067380	0.066104	0.051789	0.043876	0.038964
m''' (fuse)/g	0.001098	0.001106	0.001017	0.001001	0.000833	0.001000	0.001056
n ⁱ (H ₂ O)/mol	0.05535	0.05535	0.05535	0.05535	0.05535	0.05535	0.05535
Δt _c /K = (t _f - t _i + Δt _{corr})/K	1.984647	1.998490	2.001331	1.999425	1.999988	1.997161	2.000148
ε (calor) (-Δt _c)/cal _{th}	-7949.54	-8004.39	-8016.37	-8008.73	-8010.99	-7999.66	-8011.63
ε (cont) (-Δt _c)/cal _{th} ^b	-9.99	-10.00	-10.01	-9.99	-9.98	-9.99	-8.80
ΔE _{ign} /cal _{th}	0.18	0.18	0.18	0.18	0.18	0.18	0.18
ΔE _{dec} (HNO ₃)/cal _{th}	0.00	0.00	9.51	0.00	0.00	0.00	0.00
ΔE _f (corr to std states)/cal _{th} ^c	3.24	3.28	3.22	3.28	3.30	3.30	3.31
{-m'' (ΔE _c ^o /m) (auxiliary oil)}/cal _{th}	871.14	758.04	741.48	727.44	569.91	482.83	428.78
{-m''' (ΔE _c ^o /m) (fuse)}/cal _{th}	4.45	4.48	4.12	4.05	3.37	4.17	4.28
{m' (ΔE _c ^o /m) (RJ-6)}/cal _{th}	-7080.52	-7249.01	-7267.87	-7283.77	-7444.21	-7519.17	-7583.88
{(ΔE _c ^o /m) (RJ-6)}/cal _{th} g ⁻¹	-10532.97	-10532.40	-10533.77	-10533.56	-10532.29	-10533.79	-10532.95
{(ΔE _c ^o /m) (RJ-6)}/cal _{th} g ⁻¹	-10533.10 ± 0.24 (mean and standard deviation of the mean)						

^a The symbols and abbreviations of this table are those of W. N. Hubbard et al, *Experimental Thermochemistry*, Chap. 5, pp. 75-128. F. D. Rossini, editor. Interscience: 1956.

^b $\epsilon^i(\text{cont}) (t_i - 298.15 \text{ K}) + \epsilon^f(\text{cont}) (298.15 \text{ K} - t_f + \Delta t_{\text{corr}})$.
form of Hubbard et al (footnote a).

^c

Item 81 to 85, 87 to 90, 93 and 94 of the computation

With the JP-10 sample, repeat measurements at a given temperature always showed a lower vapor pressure than had been observed earlier. The last determination was made at 278.15 K and showed the vapor pressure to be only 57 percent of that observed early in the study at that same temperature.

All of the data obtained are shown graphically in Figure 1, with the sequence in which the data points were obtained. These data at least show the general magnitude of the vapor pressure of JP-10 over a temperature range on each side of room temperature, even if they are not suitable for representing by an empirical equation or assigning precise numerical values.

5. COMBUSTION CALORIMETRY OF HYDROGENATED DIMERS OF NORBORNADIENE AND TETRAHYDRODICYCLOPENTADIENE

Experimental measurements are in progress of the enthalpies of combustion of hexacyclic *exo,exo*-dihydrodinorbornadiene and hexacyclic *endo,endo*-dihydrodinorbornadiene, compounds 1 and 3 of Table 1, and of a newly purified sample of tetrahydrodicyclopentadiene (compound 4 of Table 1). All of these materials were supplied by Professor C. Moynihan of Catholic University of America.

6. SYNTHESIS AND PURIFICATION OF ALKYLNAPHTHALENES AND ALKYLINDANS

Current work involves the synthesis of hydrocarbons 6 and 10 of Scheme I and 18, 20, 22, 25, 27 and 29 of Scheme II as well as 32 and 35 of Scheme III.

Progress in preparing hydrocarbons 6 and 10 of Scheme I has been delayed because of difficulty in hydrogenolyzing the carbon-oxygen bond of 2 which leads to 3. Subsequently, a practical low-pressure hydrogenation procedure was found which provides 3. However, since 3 is partially hydrogenated it becomes necessary to aromatize to the fully aromatic naphthalene system. Ordinarily this is not a problem, but in this case, loss of side chain during dehydrogenation has been experienced. As a result, considerable 7 is formed in preparing 6. The presence of 7 complicates the purification of 6. Consequently, the alternate route via 9 is being explored in an attempt to avoid side-chain cleavage.

Better success has been experienced with the reactions of Scheme II. A good supply of 14 is on hand. Since 14 is well separated from its isomers in both liquid chromatography and gas chromatography, it is believed that hydrocarbons 18, 20 and 22 can be prepared free of isomers.

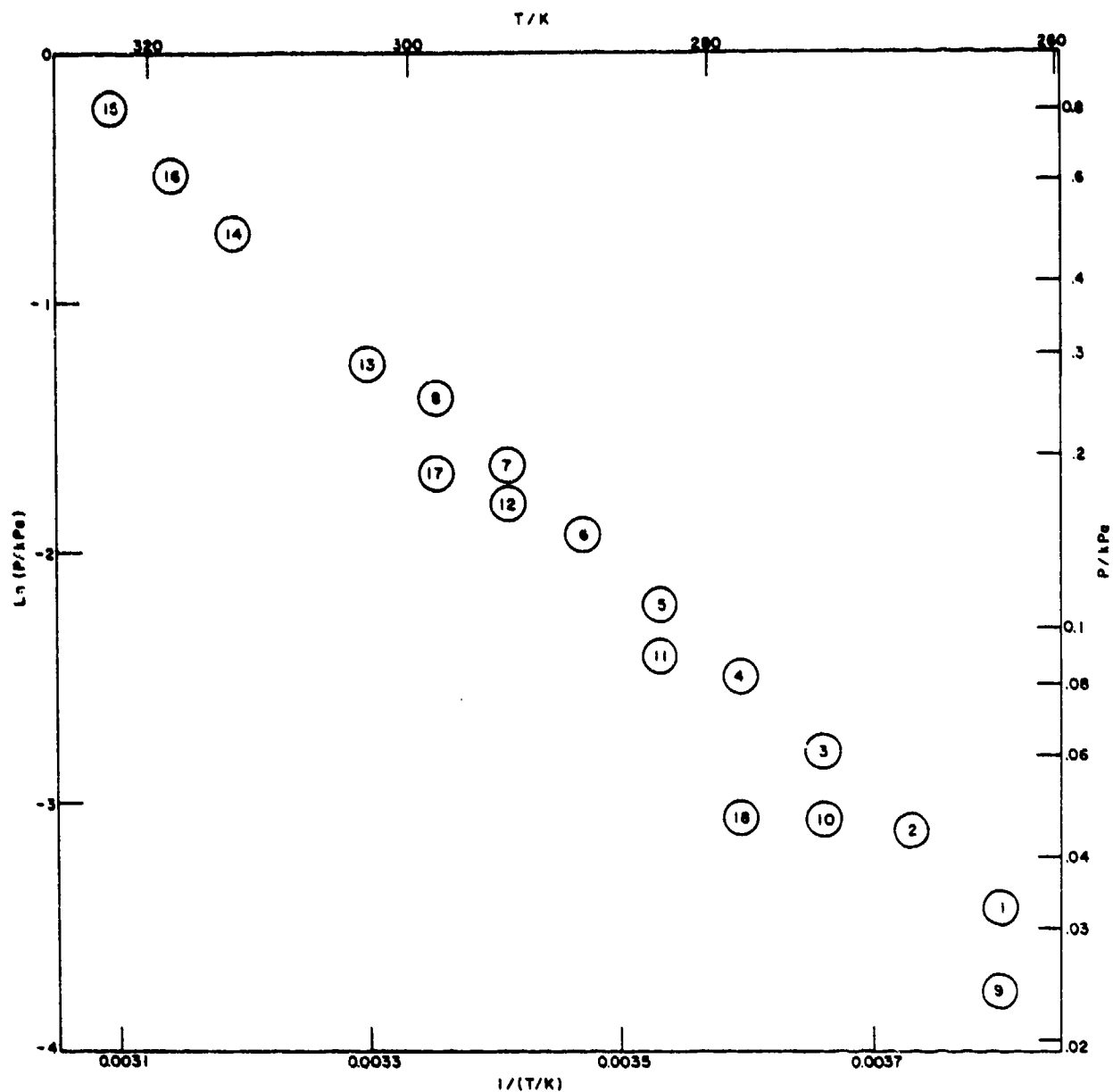
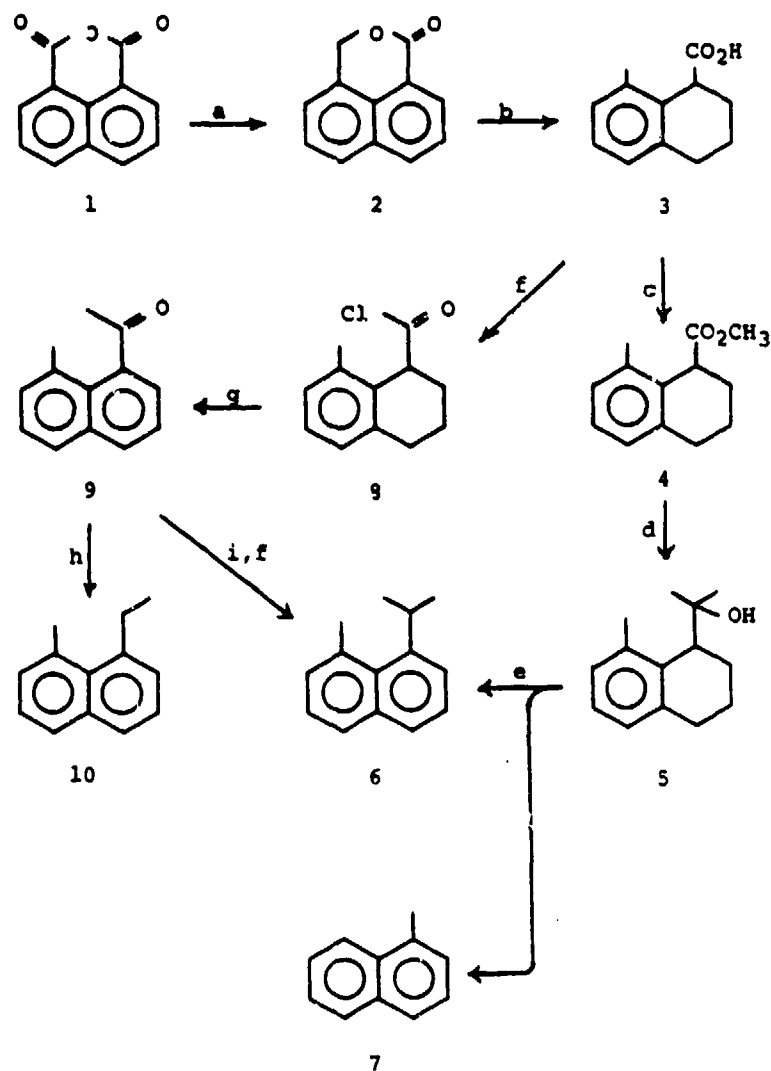


FIGURE 1. Measured values of vapor pressure of JP-10 at various temperatures, plotted as $\ln P$ versus $1/T$. The sequence in which the data points were obtained is shown by the numbering of the points.

Ketone 15 also results from the preparation of 14, and it may also be useful. However, the purification of 15 is less certain than that of 14, and consequently it may be easier to prepare acid 36 and ketone 37 as a substitute series. Both 36 and 37 respond nicely to purification.

Hydrocarbon 31 of Scheme III has been prepared, and it is anticipated that the photocyclization to 32 will be successful. Ordinarily the synthesis of 32, because of the specific placement of methyl groups, would be a formidable task, and consequently we are anxiously awaiting the result.

Scheme I



a $(i\text{-Bu})_2\text{AlH}, \text{H}_3^+\text{O}.$

b $\text{Pd/C}, \text{H}_2, \text{CH}_3\text{CO}_2\text{H}.$

c $\text{CH}_2\text{N}_2.$

d $\text{CH}_3\text{Li}, \text{ether}.$

e $\text{Pd/C}, \Delta.$

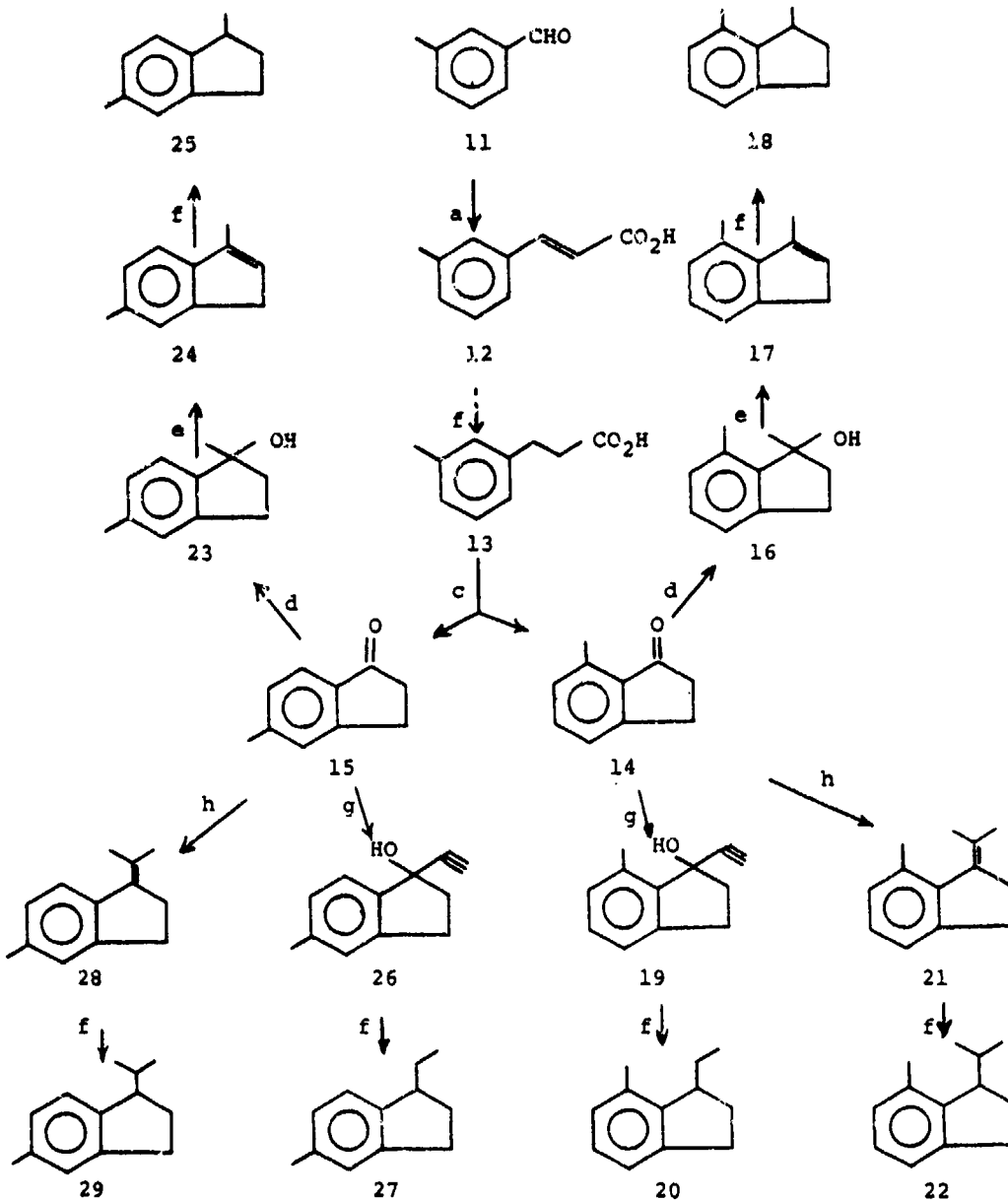
f $\text{SOCl}_2, \text{C}_6\text{H}_6, \Delta.$

g $(\text{CH}_3)_2\text{Cd}, \text{ether}.$

h $\text{NH}_2\text{NH}_2, \text{OH}^-, \text{glycol}, \Delta.$

i $\text{CH}_3\text{Li}, \text{ether}.$

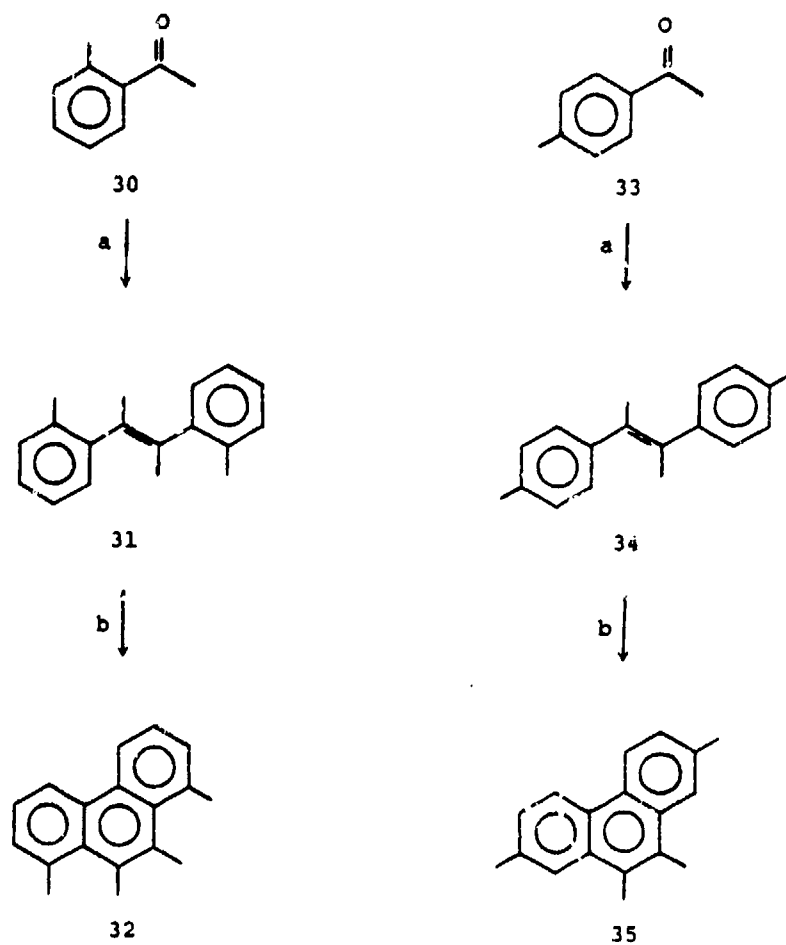
Scheme II



a Malonic acid, piperidine, pyridine. b Pd/C, H₂, acetic acid.
c PPA, Δ. d CH₃Li, ether. e Oxalic acid, ethanol, 2,4-DNPH.
f Pd/C, ethanol. g HC ≡ CNa, THF. h TiCl₃, Zn Cu,

$$\text{CH}_3\text{O}-\text{CH}_2\text{CH}_2\text{O}-\text{CH}_3.$$

Scheme III



^a $\text{TiCl}_3 \cdot \text{Zn Cu}, \text{CH}_3\text{OCH}_2\text{CH}_2\text{OCH}_3$.

^b $h\nu, \text{I}_2$.

7. PUBLICATIONS AND PRESENTATIONS

Enthalpies of Combustion of Ramjet Fuels by N. K. Smith and W. D. Good, *American Institute of Aeronautics and Astronautics Journal*, 17, No. 8, 905-907 (1979).

Enthalpy of Combustion of RJ-6 by N. K. Smith. AFOSR Special Report, AFOSR-TR-79-0508, April 1979, 5 pp.

Thermodynamics of Organic Compounds presented by W. D. Good, 1978 AFOSR Contractors Meeting on Air-Breathing Combustion Dynamics, Dayton, Ohio, Oct. 10-13, 1978.

General Techniques for Combustion of Liquid/Solid Organic Compounds by Oxygen Bomb Calorimetry by Arthur J. Head, William D. Good, and Cornelius Mosselman, Chap. 8;

Combustion of Liquid/Solid Organic Compounds with Non-Metallic Hetero-Atoms by Arthur J. Head and William D. Good, Chap. 9; in *Experimental Chemical Thermodynamics*, Volume I. Combustion Calorimetry, Pergamon Press, 1979. Prepared under auspices of International Union of Pure and Applied Chemistry, Physical Chemistry Division.

8. MANUSCRIPTS ACCEPTED FOR PUBLICATION

Thermodynamic Properties of Cyclopropylamine, Cyclopentylamine and Methylenecyclobutane by H. L. Finke, J. F. Messerly and S. H. Lee-Bechtold. Accepted by *Journal of Chemical Thermodynamics*.

Vapor Pressure of 17 Miscellaneous Organic Compounds by A. G. Osborn and D. W. Scott. Accepted by *Journal of Chemical Thermodynamics*.

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