Table III. Summary of Combustion Experiments[a] for *N,N'*-bis(*p*-methoxyphenyl)terephthalamide

m' (compd), g	0.93604	0.93577	0.93732	0.93679	0.93989
m''' (fuse), g	0.00402	0.00396	0.00425	0.00401	0.00414
Δt_c, deg	2.73992	2.73811	2.74430	2.74282	2.75269
n^i (H_2O), mole	0.05468	0.05418	0.05540	0.05529	0.05595
ϵ (calor) ($-\Delta t_c$), cal	−6535.15	−6530.83	−6545.59	−6542.06	−6565.61
ϵ (cont) ($-\Delta t_c$), cal[b]	−12.38	−12.35	−12.43	−12.45	−12.54
ΔE, cor. to std. states, cal	4.51	4.51	4.56	4.55	4.57
ΔE^fdec (HNO_3), cal	9.80	10.04	12.90	12.00	11.80
$-m''' \Delta Ec°/M$ (fuse), cal	16.57	16.32	17.51	16.52	17.06
ΔE_{Ign}, cal	0.32	0.64	0.66	0.52	0.64
$\Delta Ec°/M$ (compd), cal g^{-1}	−6961.59	−6958.62	−6958.55	−6960.92	−6962.60
Mean value and std. dev. of mean		−6960.46 ± 0.81			
Derived results at 298.15K kcal mol^{-1}					
$\Delta Ec° = -2620.03 \pm 0.76$					
$\Delta Hc° = -2621.21 \pm 0.76$					
$\Delta Hf°$ (c) $= -131.06 \pm 0.80$					
$\Delta H_{sub} = 54.4 \pm 2.0$					
$\Delta Hf°$ (g) $= -76.6 \pm 2.2$					

[a] Uncertainty interval is taken as twice the final overall standard deviation (*10*). Reaction temperature is 298.15K. Symbols and terminology are those of ref. *6*. [b] ϵ^i (cont) $(t_i - 25°) + \epsilon^f$ (cont) $(25° - t_f + \Delta t_{cor})$.

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Heat of Combustion of 3-Amino-5-methylisoxazole

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The heat of combustion of crystalline 3-amino-5-methylisoxazole in the standard state at 25°C, $\Delta Hc°$ is -566.37 ± 0.13 kcal mol^{-1}. With use of appropriate auxiliary data, this gives $\Delta Hf°$ (c) $= -14.78 \pm 0.14$ kcal mol^{-1} and $\Delta Hf°$ (g) $= 5.0 \pm 0.6$ kcal mol^{-1}.

In continuation of a study of the thermodynamic properties of isoxazoles and related compounds, we have measured the heat of combustion of 3-amino-5-methylisoxazole. The heat of combustion of this compound has not been previously reported in the literature.

Experimental

Apparatus and procedures. The combustion experiments were carried out in a Parr Instrument Co. Series 1300 oxygen bomb calorimeter with the outer jacket temperature controlled by a Hallikainen Thermotrol to ±0.002°C at 28°C. The samples were burned in a Parr Instrument Co. 1106 bomb having an internal volume of 342 ml which was initially filled with oxygen at 30 atm. Matheson ultrahigh-purity grade oxygen was used without further treatment. The sample pellets were weighed to an accuracy of 0.01 mg and corrections for air buoyance applied. The temperature of the water in the calorimeter bucket was measured with a Hewlett-Packard Model 2801-A quartz thermometer connected to a digital recorder.

One gram of water was placed in the bomb, and the initial temperature of the calorimeter was adjusted with a built-in heater to 24.45°C. The temperature was then allowed to drift to 24.55°C, at which time the experiment was begun. The temperature of the calorimeter was printed every 10 sec to the nearest 0.0001°C. Each time a print was made, an electrical pulse was registered on an electromechanical counter; when the 75th pulse was registered, a switch on the counter closed the ignition circuit firing the bomb.

Ignition was accomplished by fusing a 10-cm length of 44-swg platinum wire wrapped around a small piece (~4 mg) of Whatman No. 1 filter paper. The heat of combus-

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Table I. Summary of Combustion Experiments[a]

m' (compd), g	1.10870	1.10872	1.11078	1.10906	1.10944
m''' (fuse), g	0.00421	0.00420	0.00417	0.00420	0.00414
Δt_c, deg	2.71228	2.71181	2.71667	2.71307	2.71448
n^i (H_2O), mole	0.05501	0.05529	0.05473	0.05512	0.05556
ϵ (calor) ($-\Delta t_c$), cal	−6430.14	−6429.02	−6440.54	−6432.01	−6435.35
ϵ (cont) ($-\Delta t_c$), cal[b]	−12.93	−12.95	−12.89	−12.90	−12.94
ΔE, cor. to std. states, cal	4.19	4.21	4.18	4.18	4.21
ΔE^fdec (HNO_3), cal	19.65	20.17	19.70	19.54	20.97
$-m''' \Delta Ec°/M$ (fuse), cal	17.35	17.31	17.18	17.31	17.06
ΔE_{Ign}, cal	0.53	0.64	0.50	0.50	0.53
$\Delta Ec°/M$ (compd), cal g^{-1}	−5773.74	−5772.10	−5772.40	−5773.70	−5773.65
Mean value and std. dev. of mean		−5773.12 ± 0.36			
Derived results at 298.15K kcal mol^{-1}					
$\Delta Ec° = -566.37 \pm 0.13$					
$\Delta Hc° = -566.37 \pm 0.13$					
$\Delta Hf°$ (c) $= -14.78 \pm 0.14$					
$\Delta H_{\mathrm{sub}} = 19.5 \pm 0.6$					
$\Delta Hf°$ (g) $= 5.0 \pm 0.6$					

[a] Uncertainty interval is taken as twice the final overall standard deviation (**7**). Reaction temperature is 298.15K. Symbols and terminology are those of ref. **3.** [b] ϵ^i (cont) ($t_i - 25°$) + ϵ^f (cont) ($25° - t_f + \Delta t_{\mathrm{cor}}$).

tion of the filter paper was taken as 4118 ± 10 cal g^{-1} (*8*). The electrical ignition energy was measured with a current integrator similar to that used by Pilcher and Sutton (*5*). The extent of combustion was based on the mass of sample. All calculations, including conversion of time and temperature measurements to initial and final temperatures, correction for heat exchange between calorimeter and jacket, and reduction to the standard state, were carried out on a digital computer (*10*). The computer program followed the procedure of Hubbard et al. (*3*).

Materials. The sample of 3-amino-5-methylisoxazole (Roche Chemical Division, Hoffmann-LaRoche Inc.) was purified by recrystallization from petroleum ether and subsequent sublimation under reduced pressure. The resulting material had a melting point of 47.6°C. Analysis of melting temperature as a function of fraction melted by use of a differential scanning calorimeter (*6*) indicated a purity of 99.76 mol % for the sample. The calorimeter was calibrated with benzoic acid, NBS Sample 39i, which had a heat of combustion of 26.434 ± 0.003 absolute kJ-g^{-1} under certificate conditions.

Results

Units of measure and auxiliary quantities. All data reported are based on the 1961 atomic weights (*1*) and the 1963 fundamental constants and definitions of the thermodynamic temperature scale and of the thermochemical calorie (*2*). For reducing weights in air to weights in vacuo and correcting to standard states, the following values were used for the properties of 3-amino-5-methylisoxazole: density 1.45 g-ml^{-1}; specific heat, 0.357 cal deg^{-1} g^{-1}; and $(\partial E/\partial P)_T$, −0.0066 cal atm^{-1} g^{-1}. The density was measured with a Fekrumeter, the specific heat was measured on a Perkin-Elmer DSC-1B differential scanning calorimeter (*4*), and the energy coefficient was estimated from the temperature dependence of density in a similar compound by using the approximation, $(\partial E/\partial P)_T = -T(\partial v/\partial T)_P$.

Calorimetric results. The apparent energy equivalent of the calorimeter (calor) was determined from 11 calibration runs. The average value was 2370.75 ± 0.22 cal deg^{-1}, where the uncertainty is expressed as the standard deviation of the mean. Five satisfactory combustion experiments were obtained. Data for the combustion experiments are summarized in Table I. These results refer to the reaction:

$$C_4H_6ON_2\ (c) + 5O_2\ (g) = 4CO_2\ (g) + 3H_2O\ (l) + N_2\ (g)$$

Derived results. Using the heat of formation of water and carbon dioxide reported in the National Bureau of Standards tables (*9*) gives $\Delta Hf°$ (c) = −14.78 ± 0.14 kcal mol^{-1}. Measurement of the heat of sublimation was made in this laboratory with the Knudson method which yielded a value of 19.5 ± 0.6 kcal mol^{-1}. This result is based on five determinations of the rate of effusion at 30° and at 20°C; both sets of experiments were carried out at a pressure of about 10^{-6} torr. The heat of sublimation was calculated from a combined form of the Clausius-Clapeyron equation. Combination of $\Delta Hf°$ (c) and ΔH subl gives $\Delta Hf°$ (g) = 5.0 ± 0.6 kcal mol^{-1}.

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