Alkenylboranes

Characterization of Methylvinylboranes

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METHYLVINYLBORANES and trivinylborane were characterized as the result of an improved preparative method (*4*) through observation of their infrared, ultraviolet, nuclear magnetic resonance (NMR), and mass spectra vapor pressures, and by vapor density. The ultraviolet and NMR spectra are published elsewhere (*4*). The gas-liquid chromatographic (GLC) properties were determined, and with suitable calibration they can be used henceforth for identification.

METHODS AND RESULTS

The GLC method previously described (*10, 12*) was modified by abandoning the recirculation of helium. Instead, the gas (Airco high purity helium) was passed into the apparatus through a column packed with Molecular Sieves (Type 5A, 1/16-inch pellets from Linde Air Products Co. Division, Union Carbide Corp.). The pressure was maintained constant by a glass-mercury regulator (*6*), and the flow rate was observed by means of a rotometer (F and P Co. precision bore Flowrator 08 - 1/16 - 08 - 4/32 with the sapphire sphere). For accurate measurements a moving soap film flowmeter (*8*) was installed at the exit. The flow rates were corrected for barometric pressure and the vapor pressure of the soap solution. The collecting system consisted of a parallel-series arrangement of cold traps packed with stainless steel wool and glass wool connected by flat-ground stopcocks (Kern-Exelo Brand distributed by Kern Laboratory Supply Co., Los Angeles 34, Calif.). The chromatographic columns were packed with Johns-Manville firebrick of 32-65 Taylor mesh impregnated with standard white oil No. 9 in a weight ratio of 0.38 to 1 applied from *n*-pentane and vacuum-dried at 80° C. for 1 hour before and after application of the oil. One column used in the purification of difluorovinylborane was constructed from 10-mm. I.D. borosilicate glass tubing filled with 50.0 grams of packing in 100-cm. length. Before use the column was conditioned by treatment with boron trifluoride at nearly 1-atm. pressure for 24 hours. Another column was constructed from 6.5-mm. I.D. borosilicate glass tubing filled with 9.85 grams of the same packing over a length of 45 cm. conditioned by a treatment with boron trichloride for 2 hours. Pertinent data are collected in Table I (*1, 7*).

Trimethylborane, the methylvinylboranes, trivinylborane, and triethylborane have specific retention volumes, V_g, which vary linearly with the molecular weight. Trivinylborane and the chlorovinylboranes form a similar linear set, but boron trichloride is slightly off the line.

The mass spectra were determined on a Consolidated Model 21-103 mass spectrometer operating at 70 volts with a rhenium filament. The measurements were made at two different pressures (about 30 and 100 microns) to assist in identification of trace impurities. These and few small background peaks were subtracted from each spectrum and by using a boron isotope ratio (*13*), 0.2500, and carbon isotope ratio, 0.01204, the results were reduced to the monoisotopic spectra recorded in Table II. The closeness of fit for a monoisotopic spectrum was judged from the residual $R = \Sigma N_i^2/\Sigma I_i$ where N_i is the residue and I_i the intensity, respectively, for the i th peak. The polyisotopic spectra and a catalog of the impurities are recorded (*5*). The polyisotopic spectra of the chlorovinylboranes are listed in Table III.

Table I. GLC Properties[a]

Compound	V_R°	H	V_g	n	s
$(CH_3)_3B$	67	16	17.5	100	558
$(CH_3)_2BC_2H_3$	201	60	63	117	517
$CH_3B(C_2H_3)_2$	697	219	232	182	418
$(C_2H_3)_3B$	2250	718	761	340	425
$(C_2H_3)_2BCl$	1377	438	464	97	383
$C_2H_3BCl_2$	551	172	182	...	...
BCl_3	243	73	77	...	...
$(C_2H_5)_3B$	4000	1280	1360	...	...
$(C_2H_3)BF_2$	154	4.4	5.1	87	...

n = column efficiency as number of theoretical plates.
s = detector sensitivity, ml. mv./mg.

[a] At 22° C., atm. pressure, except $(C_2H_3)BF_2$ which was determined at 0°. V_R° = corrected retention volume in cc. H = partition coefficient. V_g = specific retention volume in cc.

Infrared measurements were made on a Perkin-Elmer Model 21 spectrometer with sodium chloride and calcium fluoride optics. All measurements were made on gas phases, and atmospheric absorption peaks were used for calibration. The wave numbers reported are believed accurate to ±5 cm.$^{-1}$. Previously unpublished infrared spectra are listed for dimethylvinylborane and methyldivinylborane in Table IV. The spectrum measured for trivinylborane was identical (within the experimental error) with that published elsewhere (*3*).

For further characterization, the standard method of static vapor pressure measurement was applied as developed by Stock (*15*) and Schlesinger and coworkers (*14*). The results are recorded in Table V, and the constants in Table VI are collected for the Antoine equation calculated from the data.

In Table VII are given the molecular weights of the methylvinylboranes and some other alkenylboranes examined by the measurements reported in this paper.

DISCUSSION

The original separation of trivinylborane by fractional condensation (*10*) gave a small amount of that product with an identity based upon complete ultimate analysis. In a later separation by the GLC method trivinylborane became confused with another substance of nearly the same GLC properties and vapor density. There seems now no doubt concerning the identity of trivinylborane as demonstrated by analyses based on instrumental measurements.

Trivinylborane and the methylvinylboranes had molecular weights corresponding to the calculated values (Table VII).

Each substance contained the assigned number of boron, carbon, and hydrogen atoms as shown by the mass spectra, because the residue from the conversion to the monoisotopic spectrum for a wrong empirical formula could not be as small as 7.7×10^{-5} found for trivinylborane, with comparable fit for the others.

Table II. Partial Monoisotopic Mass Spectra of Vinylboranes

Ion Species	Relative Intensities: Trivinylborane +	Trivinylborane 2+	Methylvinylborane +	Methylvinylborane 2+	Dimethylvinylborane +	Dimethylvinylborane 2+
BC_6H_9	0.76	0.06	...	...	...	...
BC_6H_8	100.0	0.25	...	...	...	...
BC_6H_7	17.2	0.55	...	...	...	...
BC_6H_6	13.5	0.13	...	...	...	...
BC_6H_5	14.9	0.16	...	...	...	...
BC_6H_4	0.97	0.07	...	...	...	...
BC_6H_3	0.11	0.76	...	...	...	...
BC_6H_2	0.63	0.24	...	...	...	...
BC_6H	0.35	0.40	...	...	...	...
BC_6	0.51	...	...	...	...	...
BC_5H_9	0.71	...	41.8	...	...	...
BC_5H_8	1.32	...	3.98	...	...	...
BC_5H_7	11.6	...	7.43	...	...	...
BC_5H_6	31.1	...	4.31	...	...	...
BC_5H_5	7.18	...	1.84	...	...	...
BC_5H_4	0.56	...	0.43	...	...	...
BC_5H_3	1.23	...	1.17	...	...	...
BC_5H_2	0.92	...	1.49	...	...	...
BC_5H	0.20	...	0.36	...	...	...
BC_5	...	...	0.11	...	...	...
BC_4H_9	...	...	...	...	10.5	...
BC_4H_8	0.68	...	...	...	0.23	...
BC_4H_7	11.4[a]	...	...	...	...	...
BC_4H_6	54.0	...	100.0	0.03	...	...
BC_4H_5	33.0	0.06	26.3	0.02	...	...
BC_4H_4	22.4	0.08	5.06	0.13	...	...
BC_4H_3	2.20	0.57	0.78	0.53	...	...
BC_4H_2	14.8	0.18	5.06	0.18	...	...
BC_4H	3.08	0.06	1.50	0.17	...	...
BC_4	0.14	0.03	...	...	...	...
BC_3H_6	9.22[a]	...	73.3	...	100.0	?
BC_3H_5	7.36[a]	...	22.5	...	0.70	?
BC_3H_4	22.8	0.03	34.7	0.07	6.85	0.35
BC_3H_3	9.67	?	21.3	?	5.47	?
BC_3H_2	3.77	0.03	11.6	0.04	3.52	0.08
BC_3H	0.95	?	2.36	?	0.77	...
BC_3	0.17	0.11	0.13	0.09	...	...
BC_2H_8	...	...	...	...	3.40[a]	...
BC_2H_7	1.77[a]	...	24.0[a]	...	20.8[a]	...
BC_2H_5	16.5[a]	...	8.80[a]	...	7.50[a]	...

Ion Species	Relative Intensities: Trivinylborane +	Trivinylborane 2+	Methylvinylborane +	Methylvinylborane 2+	Dimethylvinylborane +	Dimethylvinylborane 2+
BC_2H_4	87.2[a]	...	48.0[a]	...	11.4[a]	0.03
BC_2H_3	12.0	0.09	4.64	...	1.85	0.01
BC_2H_2	90.5	0.01	44.5	...	...	0.01
BC_2H	4.44	...	3.83	...	...	...
BC_2	1.10	0.02	0.75	...	...	...

Difluorovinylborane: Ion Species	Relative Intensities +
$BC_2H_3F_2$	100.0
$BC_2H_2F_2$	18.0
BC_2HF_2	15.9
BC_2F_2	0.79
$BCHF_2$	1.37
BF_2	78.1
BC_2H_3F	31.0[b]
BC_2H_2F	21.8
BC_2HF	43.6
BC_2F	7.92
BCH_3F	1.34
BCH_2F	0.67
BCHF	2.25
BCF	1.18
BHF	14.9
BF	11.5
BC_2H_2	1.02
BC_2H	1.17
BC_2	0.96

Chloromethylbinylborane: Ion Species	Relative Intensities +
BC_3H_6Cl	51.1
BC_3H_5Cl	0.2
BC_2H_3Cl	100.0
BC_2H_2Cl	2.0
BC_2HCl	9.7
BCH_3Cl	40.7
BCH_2Cl	33.5
BCHCl	4.0
BHCl	25.4
BCl	1.6
BC_3H_6	65.0
BC_3H_5	2.2
BC_3H_4	12.8
BC_3H_3	12.1
BC_3H_2	11.3
BC_3H	3.0
BC_2H_4	5.8
BC_2H_3	0.2
BC_2H_2	10.1
BC_2	1.1
HCl	47.9

[a] Species generated by elimination or migration. [b] 2+, 1.36.

Table III. Polyisotopic Spectra

m/e[a]	Intensity	m/e	Intensity	m/e	Intensity
(Dichlorovinylborane Ionizing potential of 8.0 volts[b])					
28	28.0	57	16.4	83	2.4
29	14.4	58	1.3	84	9.3
30	2.9	59	0.7	85	0.8
31	5.1	60	13.5	88	1.4
35	1.0	61	44.8	89	2.2
36	485.	62	4.6	90	7.8
37	125.3	63	13.8	91	2.0
38	139.6	64	2.5	92	6.8
39	41.0	65	0.8	93	0.8
40	1.3	67	8.9	94	1.6
41	17.3	68	3.0	99	0.8
42	11.1	69	10.7	100	2.2
43	5.9	70	4.9	101	0.5
44	1.8	71	4.7	102	0.8
45	0.8	72	41.3	107	11.4
46	2.6	73	100.	108	43.0
47	7.2	74	18.5	109	8.1
48	0.5	75	33.9	110	26.0
49	2.1	76	5.7	111	1.9
52	0.7	77	2.0	112	4.4
53	3.2	80	0.9	113	0.8
54	11.3	81	3.6		
55	33.7	82	1.4		
56	19.7				

m/e[a]	Intensity	m/e	Intensity	m/e	Intensity
(Chlorodivinylborane Ionizing potential of 70 volts[c])					
28	28.7	53	13.4	75	0.83
29	23.3	54	19.0	76	1.36
30	10.2	55	37.5	77	1.97
31	14.9	56	28.6	78	1.99
32	0.76	57	31.2	79	2.39
35	64.6	58	3.57	80	2.49
36	208.1	59	0.99	81	6.79
37	108.4	60	2.34	82	17.1
38	64.5	61	5.64	83	15.5
39	72.8	62	3.47	84	15.8
40	3.06	63	9.65	85	1.04
41	19.9	64	28.5	86	0.08
42	4.43	65	100.0	87	0.03
43	5.27	66	5.56	89	0.02
44	4.82	67	14.4	90	4.60
45	7.68	68	1.90	91	7.01
46	0.69	69	3.13	92	10.36
47	1.06	70	0.89	93	1.60
48	1.31	71	3.27	99	0.27
49	2.51	72	1.01	100	0.64
50	4.14	73	1.58	101	0.20
...	...	74	0.71	102	0.64

(Continued)

Table III. Polyisotopic Spectra (*Continued*)

(Trivinylborane Sensitivity at 70 volts 20.8 div./μ for m/e) (adjusted to butane sensitivity of 50 for m/e 43)

m/e[a]	Intensity	m/e	Intensity	m/e	Intensity
12	3.94	45	0.56d	78	13.4
13	10.5	46	0.06d	79	2.11
14	3.65	47	0.40	80	0.90
15	3.23	48	1.87	81	1.08
16	0.12	49	6.12	82	2.34
17	...	50	15.2	83	0.60
18	...	51	24.6	84	0.53
19	0.05	52	10.3	85	0.67
20	...	53	9.88	86	0.39
21	0.40	54	3.77	87	1.33
22	0.04	55	5.24	88	4.80
23	0.12	56	0.87	89	17.6
24	0.76	57	0.22	90	42.2
25	2.73	58	0.09	91	100
26	14.2	59	0.99	92	7.40p
27	25.9	60	6.64	93	0.35
28	1.20	61	15.3	94	0.08
29	4.00	62	8.27	95	0.05
30	0.20d	63	30.3	96	...
31	0.59d	64	46.7	97	...
32	0.06d	65	57.5	98	...
33	0.04	66	13.8	17.5	0.02d
34	0.24	67	1.21	18.5	0.01d
35	2.18	68	0.15	23.5	0.12d
36	26.6	69	0.15	24.5	0.04d
37	92.3	70	0.13	25.5	0.03d
38	35.1	71	0.11	30.5	0.20d
39	90.2	72	0.42	31.5	0.08d
40	18.2	73	1.21	42.5	0.26d
41	2.20	74	1.44	43.5	0.10d
42	0.59d	75	2.38	44.5	0.19d
43	0.80d	76	14.6	45.5	0.25d
44	0.30d	77	33.9	22.7	0.03m

(Methyldivinylborane Sensitivity at 70 volts 20.41 div/μ for m/e) (65 adjusted to butane sensitivity of 50 for m/e 43)

12	3.38	39	49.7	66	4.85
13	7.30	40	15.7	67	1.63
14	4.86	41	24.2	68	0.18
15	10.4	42	3.10	69	0.54
16	0.58	43	4.58	70	1.41
17	0.32	44	0.60	71	0.25
18	1.03	45	0.09	72	0.71
19	0.05	46	0.07	73	0.76
20	...	47	0.71	74	1.36
21	0.12	48	5.17	75	0.95
22	0.04	49	16.8	76	2.89
23	0.20	50	30.0	77	6.17
24	1.19	51	40.6	78	8.56
25	7.09	52	41.4	79	14.5
26	42.3	53	76.9	80	41.6 p
27	97.4	54	19.2	81	2.39
28	1.08	55	9.68	82	0.15
29	3.05	56	0.74	83	0.09
30	0.17d	57	0.28	84	0.06
31	0.53d	58	0.09	85	0.01
32	0.02d	59	0.53		
33	0.05	60	2.85	22.7	0.03m
34	0.23	61	5.79	23.5	0.09d
35	1.68	62	2.24	24.5	0.04d
36	14.7	63	11.5	25.5	0.07
37	45.2	64	50.6	30.5	0.18d
38	17.4	65	100.0	31.5	0.13d

d = doubly charged ion p = parent peak
m = metastable peak r = rearrangement peak

[a] Mass per unit of electronic charge. [b] Spectrum adjusted to 100 for m/e 73 rather than m/e 36 as this peak is due to HCl^-. [c] Spectrum adjusted to 100 for m/e 65 rather than m/e 36.

(Dimethylvinylborane Sensitivity at 70 volts 49.31 div./μ for m/e) (53 adjusted to butane sensitivity of 50 for m/e 43)

m/e[a]	Intensity	m/e	Intensity	m/e	Intensity
12	1.94	35	0.73	61	1.04
13	4.68	36	5.01	62	0.17
14	2.75	37	14.2	63	0.34
15	14.9	38	4.99	64	0.17
16	0.48	39	13.3	65	0.19
17	0.01	40	12.9	66	0.13
18	0.01d	41	21.1	67	2.83
19	0.03d	42	1.78	68	10.53p
20	...	43	3.43	69	0.53
21	...	44	0.24	70	0.06
22	0.02	45	0.18	71	0.03
23	0.13	46	0.03	72	0.04
24	0.71	47	0.24	13.8	0.15m
25	3.07	48	1.64	18.5	0.02d
26	16.8	49	4.88	19.5	0.03d
27	37.8	50	7.29	23.6	0.08d
28	2.82	51	7.20	24.5	0.08d
29	1.74	52	25.7	25.5	0.35d
30	0.09d	53	100.0	26.5	*d
31	0.57d	54	3.57	30.5	0.53d
32	0.07d	55	0.98	31.5	0.24d
33	0.01d	56	4.35	32.5	0.05d
34	0.09d	57	16.5	33.5	0.06d
		58	0.42		
		59	0.22		
		60	0.75		

(Difluorovinylborane Sensitivity at 70 volts 11.57 div./μ for m/e) (76 adjusted to butane sensitivity at 50 for m/e 43)

28	6.72	46	0.31	64	0.17
29	3.93	47	0.93	65	0.18
30	15.1	48	19.8	66	0.05
31	14.8	49	77.8	67	0.03
32	0.33	50	0.06	68	0.03
33	0.24	51	0.31	69	0.06
34	0.25	52	0.08	70	0.07
35	1.25	53	2.21	71	0.03
36	1.44	54	18.7	72	0.25
37	1.03	55	49.0	73	4.74
38	0.12	56	30.3	74	20.4
39	0.27	57	31.3	75	43.1
40	0.27	58	0.84	76	100.0 p
41	1.39	59	0.23	77	2.38
42	1.75	60	0.11	78	0.13
43	2.42	61	0.42	28.5	1.36d
44	1.03	62	1.40	40.4	0.17m
45	1.34	63	0.19	41.4	0.31m

(Chloromethylvinylborane Sensitivity at 70 volts 17.5 div./μ for) (m/e 73 adjusted to butane sensitivity of 50 for m/e 43)

13	3.89	43	7.00	73	100.0
14	4.40	44	2.01	74	10.2
15	8.62	45	2.62	75	31.4
16	0.22	46	7.62	76	0.74
17	...	47	25.2	77	...
18	0.16	48	8.33	78	0.50
19	0.10	49	20.9	79	...
20	0.04	50	15.0	80	0.25
21	...	51	13.2	81	0.20
22	0.11	52	18.0	82	0.73
23	0.69	53	62.5	83	1.94
24	2.30	54	3.17	84	1.76
25	6.55	55	1.16	85	1.00
26	21.5	56	1.40	86	0.67
27	41.4	57	5.58	87	12.6
28	5.12	58	0.14	88	49.5
29	1.28	59	11.9	89	5.68
30	0.42	60	42.2	90	16.0
31	1.20	61	43.0	91	0.85
32	0.19	62	14.0	92	0.22
33	...	63	13.5	93	0.22
34	0.28	64	...	94	0.93
35	12.6	65	2.94	95	1.87
36	49.8	66	0.21	96	0.14
37	13.5	67	0.08		
38	16.6	68	0.25	17.5	1.37
39	5.61	69	0.83	18.5	0.44
40	0.65	70	3.31	24.5	0.15
41	3.33	71	9.73	25.5	0.28
42	2.00	72	27.9		

Table IV. Infrared Spectra

($Cm.^{-1}$)

Dimethylvinylborane: 3058 (m. sh.), 2959 (m.s.), 2899 (m.), 1949 (w.), 1603 (m.s.), 1414 (v.s.), 1290 (v.s.), 1206 (m.), 1170 (v.s.), 1120 (v.s.), 1015 (m.), 988 (m), 973 (s), 917 (m.), 847 (b.m.w.)

Methyldivinylborane: 3067 (m.), 3040 (m.), 2967 (m.s.), 2874 (m.w.), 1949 (m.w.), 1605 (s.), 1420 (v.s.), 1299 (s.), 1196 (m.), 1176 (s.), 1127 (m.), 1094 (s.), 1016 (s.), 972 (v.s.), 855 (b.m.)

Trivinylborane: 3078 (m.s.), 3048 (m. sh.), 2999 (m.s.), 2973 (m.s.), 1942 (m.w.), 1613 (s. sh.), 1605 (s.), 1462 (s. sh.), 1422 (v.s.), 1302 (m.w.), 1183 (m. sh.), 1153 (s.), 1119 (m. sh.), 1093 (s.), 1018 (s. sh.), 972 (v.s.), 907 (m.s. sh.), 821 (b.v.w.), 705 (b.m.)

w = weak
m = medium
s = strong
v = very
sh. = shoulder
b = broad

Table V. Vapor Pressures

t, ° C.	P_{mm}, Obsd.	P_{mm}, Calcd.
	(Difluorovinylborane)	
−81.5	62.7	62.7
−70.5	134.4	135.6
−64.2	202.7	202.0
−54.1	360.8	360.8
−51.8	407.2	407.9
−45.5	562	562
−40.8	703	703
	(Dichlorovinylborane)	
−40.1	13.7	13.7
−23.5	38.0	36.8
−7.4	83.0	85.5
−4.0	100.5	100.8
0.0	121.8	121.8
	(Chlorodivinylborane)	
−6.0	26.8	27.1
0.0	37.9	37.9
5.5	50.3	50.6
12.2	71.0	70.8
13.8	75.9	76.5
19.8	102.2	101.2
23.7	120.2	120.4
	(Dimethylvinylborane)	
−81.9	3.2	3.1
−64.1	12.7	12.7
−45.5	42.9	42.9
−23.0	145.3	146.1
0.0	411.5	411.5
6.1	524.6	525.4
	(Methyldivinylborane)	
−45.5	6.9	6.7
−22.9	27.9	28.5
0.0	95.6	95.8
10.1	153.3	153.1
14.4	184.4	184.0
21.9	253.4	253.4
	(Trivinylborane)	
−22.9	6.8	6.3
0.0	24.4	24.3
6.2	33.5	33.6
9.9	40.0	40.4
14.3	49.9	50.1
20.0	65.6	65.6
	(Methylchlorovinylborane)	
−64.0	3.5	3.4
−45.4	12.9	13.2
−38.2	19.7	19.7
−23.0	51.3	51.0
0.0	161.7	160.8
6.2	211.9	211.9

Each substance gave a strong infrared band in the carbon double bond region (*2*).

For trivinylborane, only the H^1 NMR multiplet (*4*) was found with chemical shift downfield to the extent characteristic of vinyl groups (*11*) and comparable to tetravinyltin (*9*). In addition, a single band in the methyl proton range of chemical shift was found for methyldivinylborane with an area ratio indicating vinyl to methyl proton ratio of 2 to 1. The corresponding area ratio for dimethylvinylborane was the expected 1 to 2.

Table VI. Constants from Vapor Pressure Data

Compound	A^a	B^a	C^a	ΔH^b	ΔS^c	B.P.^{d}
$C_2H_3BF_2$	10.4642	1430.0	0.006300	4.96	21.2	−39.1
$C_2H_3BCl_2$	7.6053	1508.0	...	6.90	21.6	46.0
$(C_2H_3)_2BCl$	11.4619	2230.0	0.006300	7.21	20.6	76.4
$(C_2H_3)_3B$	8.2833	1810.0	0.001000	7.69	21.4	85.7
$(C_2H_3)_2BCH_3$	8.4126	1660.0	0.001300	6.97	21.5	51.7
$(CH_3)_2BC_2H_3$	11.9166	1890.0	0.008700	5.29	18.2	17.1
$(CH_3)(C_2H_3)BCl$	8.1529	1550.0	0.001000	6.64	21.2	39.3

[a] Constant for Antoine equation $\log_{10} P$ mm. Hg $= A - B/T - CT$.
[b] Heat of vaporization kcal./mole at b.p.
[c] Entropy of vaporization at b.p., Trouton's constant.
[d] Extrapolated from vapor pressure curve.

Table VII. Molecular Weight Data

Compound	Molecular Weights		
	a	*b*	*c*
$C_2H_3BF_2$			
$C_2H_3BCl_2$	76.3	...	75.9
$(C_2H_3)_2BCl$	109.9	...	108.7
$(C_2H_3)_3B$	101.0	...	100.4
$(C_2H_3)_2BCH_3$	93.8	92	92.0
$(CH_3)_2B(C_2H_3)$	80.6	80	79.9
	68.7	68	67.9

From: *a* vapor density *b* monoisotopic mass spectrum *c* calculated.

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