TABLE VIII

SHORTEST INTER- AND INTRAIONIC NONBONDED CONTACTS (Å)[a]

Interionic[b]	
38i–63	1.8 (3)
50–52	2.0 (2)
46s–51	2.0 (2)
40i–52	2.0 (2)
48i–66	2.0 (3)
35s–64	2.1 (2)
33i–72	2.1 (3)
48i–59	2.2 (3)
32s–51	2.2 (2)
36s–51	2.2 (2)
34i–58	2.2 (3)
34i–65	2.2 (3)
8i–63	2.2 (2)
36s–62	2.2 (3)
34i–66	2.3 (2)
37i–58	2.3 (3)
40i–58	2.3 (2)
35–67	2.3 (2)
32s–74	2.3 (2)
42s–51	2.3 (2)
37i–63	2.3 (3)
47i–58	2.3 (3)
Intraionic	
Triethylammonium[c] C or N to H	
26–70	1.6 (2)
21–61	1.8 (2)
27–77	1.8 (2)
Triethylammonium C or N to H	
27–78	1.9 (2)
23–69	1.9 (1)
21–53	1.9 (2)
25–68	2.0 (2)
25–69	2.0 (2)
21–54	2.0 (2)
30–57	2.0 (2)
21–60	2.0 (2)
Intraionic	
Methylene[d] H to methyl or ammonium H	
59–72	1.5 (3)
62–68	1.9 (3)
54–66	2.1 (2)
54–69	2.1 (2)
58–61	2.1 (3)
61–63	2.1 (3)
62–78	2.1 (3)
Methyl and Methylene Group Internal Contacts[e]	
70–72	1.2 (3)
64–68	1.4 (2)
74–78	1.4 (2)
52–53	1.6 (3)
71–73	1.6 (2)
74–75	1.6 (2)
69–71	1.6 (2)

[a] Atoms transformed by symmetry operations are labeled with i for inversion or s for screw axis. [b] Estimated errors are in parentheses. The sum of van der Waals radii for H + H is about 2.4 Å. [c] The nonbonded C–H distance in staggered ethane is 2.2 Å. [d] The smallest H–H separation between methyl groups in staggered ethane is 2.5 Å. [e] In methane H–H is 1.8 Å.

nates and depend on guesses for unrefined hydrogens. Thus they are not standard deviations in the usual sense. Shortest contacts in each category involve hydrogens 63, 70, and 72, all of which were difficult to locate and occur at population 0.4. Other short interionic contacts may result from errors in H(52) and H(66) and from the fact that H(51), H(64), H(69), and H(74), though common to both disorder forms, need not be exactly superimposed. Since it is not possible to remove all short interionic contacts by an assumption of local ordering, the disorder is probably statistical in nature but with perhaps some short-range order. This conclusion is partly supported by a very slight amount of diffuseness along the *b* direction in the most intense reflections. For crystal structure studies requiring a positive ion approximating the size and shape of triethylammonium a better choice might be the N-ethylpyrrolidinium ion, in which two of the groups bonded to nitrogen are firmly tied together and therefore immune from this disorder.

Acknowledgments.—Nearly all computations of diffractometer settings, structure factors, least-squares refinements, Fourier maps, and bond distances and angles were done with the CRYRM crystallographic system[15] as modified by G. N. Reeke, Jr. The research was supported in part by the Office of Naval Research, the Advanced Research Projects Agency, and the National Science Foundation, who sponsored a predoctoral fellowship for C. H. S. We also wish to thank Al Powder for assistance in the calibration of the unit cell parameters.

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Crystal Structure of Tris(triethylammonium) μ-Nitrosobis(nonahydrodecaborate). The Structure of μ-Nitroso(nonahydrodecaborate)(3−)

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The $B_{20}H_{18}NO^{3-}$ anion, μ-nitrosobis(nonahydrodecaborate)(3−), consists of two $B_{10}H_9$ units each joined *via* an apical boron to the nitrogen atom of a bridging NO group. The approximate symmetry of this anion is C_{2v}. Some delocalization of electrons is suggested by the B–N bond distances of 1.51 (3) and 1.46 (3) Å and by the N–O distance of 1.28 (2) Å. A twofold disorder is present in one triethylammonium ion, which has an idealized nearly flat conformation of C_3 symmetry. The two ordered triethylammonium ions each differ from C_3 symmetry by rotation of one ethyl group about a C–N bond. The monoclinic space group is $P2_1/n$, and there are four formula weights in a unit cell having parameters a = 24.44 (4), b = 16.68 (3), c = 9.65 (2) Å, β = 97.0 (2)°, ρ_{obsd} = 0.99 (2) g cm^{-3}, and ρ_{calcd} = 0.972 (4) g cm^{-3}. Nearly complete refinement has yielded $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ = 0.14 for the 4555 X-ray maxima. The crystals are intensely purple, and the two visible absorption maxima are shifted toward the red in less polar solvents.

Polyhedral $B_{10}H_{10}^{2-}$ ion[1] undergoes oxidative coupling with NO_2 alone or with NO in the presence of Fe^{3+} to yield a product initially formulated[2] as $B_{14}H_{12}NO^{2-}$ but later shown[3] to be $B_{20}H_{18}NO^{3-}$. Its infrared ab-

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sorption at 1180 cm^{-1} attributed[4] to NO is considerably different from the 1385 cm^{-1} observed[5] in 1,10-$(CH_3)_2$-$SB_{10}H_8NO^-$ ion. Thus a bridging NO group can be suggested for $B_{20}H_{18}NO^{3-}$, analogous in a formal way[3] to the bridging hydrogen in $B_{20}H_{19}^{3-}$. Reduction of $B_{20}H_{18}NO^{3-}$ under mild conditions yields[4] $B_{20}H_{18}$-NH_2^{3-}, which is weakly basic, in contrast to the strongly basic B_{10} amines,[6] such as 2-$B_{10}H_9NH_2^{2-}$. This comparison would be consistent with the secondary ammonium ion formulation for $B_{20}H_{18}NH_2^{3-}$. The original suggestion[3] of the bridging of $B_{10}H_9$ units by NO in $B_{20}H_{18}NO^{3-}$ was based upon the valence theory of boron hydrides, and the expectation that the mild conditions of the oxidative coupling reactions did not destroy the B_{10} cages. This structure is confirmed by the X-ray diffraction results described below, which indicate that the N atom is bonded to a B atom linked to four other B atoms in each of the B_{10} cages.

Experimental Section

Purple crystals of $((C_2H_5)_3NH)_3B_{20}H_{18}NO$ kindly supplied by M. F. Hawthorne were used as received. The monoclinic crystals take the form of brittle plates elongated along a and c with unit cell dimensions $a = 24.44$ (4), $b = 16.68$ (3), $c = 9.65$ (2) Å, and $\beta = 97.0$ (2)°, where parentheses indicate the estimated error in the last place. Cell parameters were averaged from values from an earlier study[7] and from an independent redetermination based on $h0l$ and $hk0$ Weissenberg films exposed at 22 (2)° with Cu Kα radiation (λ 1.5418 Å) and calibrated by Al powder (cubic, $a = 4.0415$ Å). If the unit cell contains four formula weights, the calculated density of 0.972 (4) g cm^{-3} agrees with the value 0.99 (2) g cm^{-3} measured by flotation in m-xylene–bromobenzene solution. Systematic absences of $h0l$ reflections with $h + l$ odd and of $0k0$ with k odd were noted on Weissenberg films of levels $hk0$–$hk6$ and $0kl$–$2kl$. The space group symmetry $P2_1/n$ specified by these absences implies that 43 nonhydrogen atoms must be located in the asymmetric unit in the absence of disorder. No symmetry is imposed on the $B_{20}H_{18}NO^{3-}$ ion or the three independent triethylammonium groups.

Of the 9101 accessible independent reflections within the Cu sphere, 6387 were measured by film or counter methods. After about 4 weeks of exposure to Ni-filtered Cu Kα radiation crystals were discarded, because the most sensitive of six check reflections decreased by about 10% in intensity after this period. Levels $0kl$–$2kl$ and $hk0$–$hk3$ were recorded from separate crystals on equiinclination Weissenberg photographs, in which six films were exposed simultaneously in a pack. Intensities were estimated by visual comparison with a series of 42 or 37 images of a representative spot exposed for time $t_n = 1.1t_{n-1}$. For the expected error in one single intensity measurement I we chose

$$\sigma(I) = [0.33I_{\min} + bI + 0.1I^2/(I_{\max} - I)^2] \times [1 + 0.25\exp(-50.0(0.5 - \sin^2\theta)^2)]$$

and propagated these error estimates through all further calculations. Here $I_{\min}$ and $I_{\max}$ are the extremes of the calibrated intensity scale and b (about 0.08) is determined empirically. Film factors were obtained for each adjacent pair of films as the weighted logarithmic average of intensity ratios, corrected for nonnormal incidence of X-rays on upper levels, averaged to give an overall film factor, and applied to bring all observations on each level to a common scale. Short, weak, diffuse streaks mostly along a^* were noted on the films, but their integrated intensities were estimated to be below errors in measurement of the associated Bragg reflections. No analysis of these diffuse streaks was made, but it is reasonable to suppose that they are associated with the disorder described below.

A Supper-Pace Buerger diffractometer was used to record intensities on levels $hk0$, $hk4$, and $hk5$ from a third crystal and on $hk0$ and $hk5$–$hk9$ from a fourth crystal. Experimental technique for the diffractometer measurements was identical with that described for the $((C_2H_5)_3NH)_2B_{20}H_{18}$ study[8] and is therefore not repeated here. All positive intensities were carried forward and assigned errors of 0.04 times the uncorrected intensity plus twice the square root of the sum of scan count and background. For large intensities this error estimate tends toward fractional error and is about half the error assigned to corresponding film data; for small intensities the error is dominated by counter statistics and is approximately equal to the errors of film data.

The four crystals used in the study had dimensions (mounting axis given first) of 0.6 × 0.9 × 0.2, 0.8 × 0.2 × 0.1, 0.6 × 0.6 × 0.1, and 0.8 × 0.2 × 0.1 mm. No absorption corrections were made because the linear absorption coefficient $\mu = 3.65$ cm^{-1} yields minimum and maximum corrections to F_{hkl}'s of 1.04–1.18, 1.02–1.04, 1.02–1.12, and 1.02–1.04 on the zero level for the four crystals, respectively, and 1.04–1.21, 1.02–1.04, 1.02–1.13, and 1.02–1.04 at an equiinclination angle of 30°. Extinction effects were also ignored because only four of the thirteen reflections with final $F_o > 100$ had $F_o < 0.90F_c$ and none had $F_o < 0.78F_c$. Lorentz and polarization corrections were applied, and a scale factor S_i was determined for each level; for each reflection H the weighted average $\bar{I}_H$ of observations I_{Hi} was taken, yielding

$$R = \sum_{H,i} |S_iI_{Hi} - \bar{I}_H| / \sum_{H,i} \bar{I}_H = 0.09$$

In order to reduce computing time only those 1872 reflections with $F_o > 8.0$ were used in early refinements. In the final refinements only the 3802 observed reflections with $F_o > 3.9$ were retained, along with 753 unobserved reflections. Initial estimates of scale and overall temperature factors by Wilson's method were refined later by least-squares procedures. Those 4555 structure factors are listed in Table I together with final calculated values (both ×10). Of the 753 unobserved reflections, 83 had $F_c > F_{\min}$, but none had $F_c > 2F_{\min}$.

Visible light absorption spectra were recorded on a Cary Model 14 spectrometer. Solutions of 5×10^{-5} M $K_3B_{20}H_{18}NO$ in 1 vol % aqueous organic solvents were prepared by adding to 1 ml of 0.005 M stock solution, made with double-distilled water, enough reagent grade solvent from a freshly opened bottle to make up 100 ml. Quartz cuvettes with 1-cm path length held the sample and the aqueous solvent reference. Light sources in irradiation experiments were (a) for approximately 5800 Å a 100-W tungsten lamp with a $K_2Cr_2O_7$ filter to remove shorter wavelengths, (b) for approximately 4000 Å a 20-W Blak-Ray long-wave uv lamp with Pyrex filter, and (c) for 3150 Å a 500-W high-pressure mercury lamp equipped with a Bausch and Lomb monochromator with bandpass 108 Å (usable wattage 100).

Structure Determination and Refinement

A satisfactory trial structure was obtained by the symbolic addition procedure[9] on the first attempt. Normalized structure factors were calculated by the usual equation

$$E^2 = F^2/\left(\epsilon \sum_{j=1}^{N} f_{jH}^2\right)$$

in which for space group $P2_1/n$ the factor ϵ is 2 for $h0l$ and $0k0$ reflections and 1 otherwise. From the 222 reflections having $|E| > 2.0$, nine were given signs, three explicitly to specify the origin and six as unknown sym-

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TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS (×10) FOR $[(C_2H_5)_3NH]_3B_{20}H_{18}NO^a$

H	L	FO	FC	H	L	FO	FC
				-10	1	100	103
••••••••••••••••				-11	1	112	-90
				12	1	175	-208
K = 0				-12	1	395	408
				13	1	88	30
6	0	196	-142	-13	1	75	-9
8	0	664	578	14	1	137	-115
10	0	174	-171	-14	1	88	57
12	0	259	251	15	1	143	85
14	0	412	373	-15	1	146	108
16	0	355	361	-16	1	62	-67
18	0	99	50	17	1	343	363
20	0	132	-52	-17	1	166	159
22	0	114	92	18	1	261	263
-3	1	1216	-1557	-18	1	198	-180
5	1	1108	-1267	21	1	116	-83
-5	1	189	-234	25	1	106	118
7	1	638	648	28	1	61	-54
-7	1	244	-289	29	1	71	56
9	1	529	-513	0	2	1396	-1387
-9	1	517	-524	2	2	686	704
11	1	257	229	3	2	720	706
-11	1	233	253	4	2	159	195
15	1	179	-192	5	2	131	-36
-15	1	96	97	6	2	198	200
17	1	250	-242	7	2	164	140
19	1	173	-119	-7	2	731	-730
23	1	132	-109	8	2	141	-120
29	1	140	-145	-8	2	383	-330
2	2	1174	-1320	9	2	92	-107
4	2	241	-303	-9	2	399	390
6	2	857	905	10	2	96	-81
8	2	481	367	-10	2	113	99
-8	2	606	596	11	2	81	79
10	2	160	179	-11	2	525	525
-10	2	871	885	12	2	143	92
12	2	400	-386	13	2	150	115
-12	2	197	-215	-13	2	137	137

TABLE I (*Continued*)

H	L	FO	FC
••••••••••••••••			
K = 8			
-1	6	178	145
-2	6	228	-254
3	6	56	62
-3	6	117	-100
-4	6	195	149
5	6	151	157
-5	6	88	-77
6	6	97	113
-6	6	321	309
7	6	118	-97
-7	6	68	-60
8	6	148	170
-8	6	128	105
9	6	53	-39
10	6	72	-70
-10	6	51	51
11	6	51	-57
-11	6	171	156
12	6	92	-118
-12	6	175	-157
13	6	42	-66
-13	6	258	247
14	6	41	-20
-14	6	53	-23
-16	6	133	-126
-17	6	52	39
18	6	65	54
-19	6	49	50
-20	6	49	9
-1	7	78	64
-2	7	144	-122
-3	7	192	175
5	7	90	86
-5	7	95	-111
6	7	65	-88
-7	7	62	48
8	7	100	106
9	7	44	63
-9	7	71	-63
10	7	98	-122
-10	7	48	57
-12	7	41	49
14	7	106	-163
-14	7	102	-98
-16	7	138	-129
-18	7	41	-62
-19	7	55	16
-20	7	107	-131
0	8	140	136
1	8	71	-58
-1	8	86	-81
2	8	51	56
-3	8	49	45
-4	8	58	-27
-5	8	52	60
7	8	59	-48
-7	8	48	-52
8	8	63	79
9	8	87	94
-9	8	41	43
-10	8	42	-25
12	8	67	105
-12	8	47	28
13	8	42	75
-13	8	107	-102
-14	8	123	121
-16	8	80	88
-17	8	75	-104
-1	9	52	55
4	9	94	-94
5	9	44	63
-6	9	41	-25
7	9	52	-51
8	9	59	-77
-8	9	73	-57
-9	9	41	54
-10	9	101	-93
-11	9	139	148
-14	9	78	89
••••••••••••••••			
K = 9			
2	0	125	-138
3	0	237	-233
4	0	217	-182
5	0	184	215
6	0	210	201
8	0	253	258
9	0	307	-318
10	0	308	337
11	0	195	-197
12	0	357	332
13	0	173	-193
14	0	112	-96
15	0	263	-242
16	0	131	-109
18	0	81	-57
19	0	159	-166
22	0	81	14
24	0	178	190
26	0	49	58
2	1	184	136
-3	1	266	226
4	1	80	88
-4	1	58	-2
5	1	348	325
-5	1	227	-179
6	1	65	1
-7	1	96	104
8	1	340	314
-8	1	245	-237
9	1	277	273
-9	1	362	352
-10	1	160	140
11	1	192	167
-11	1	171	150
-12	1	103	-76
13	1	463	442
-13	1	151	147
14	1	120	79
-14	1	104	103
-15	1	258	220
16	1	145	154
-16	1	211	-199
-17	1	120	100
18	1	91	86
-18	1	109	-54
19	1	65	74
20	1	67	53
21	1	86	88
-22	1	71	87
23	1	108	-127
-23	1	107	-95
0	2	126	89
-2	2	170	-163
-3	2	83	-86
-4	2	185	-165
5	2	230	-219
-5	2	170	-133
6	2	148	-160
-6	2	342	323
7	2	166	-161
8	2	226	-199
-8	2	169	174
9	2	75	70
-9	2	83	-89
10	2	81	77
-10	2	144	129
11	2	204	173
-11	2	225	202
-12	2	275	279
13	2	49	46
-13	2	102	133
-14	2	153	157
15	2	90	110
-15	2	147	158
16	2	137	106
-16	2	161	-163
-18	2	127	-115
-19	2	78	35
22	2	63	43
23	2	96	-93
-23	2	73	-68
0	3	104	-67
1	3	85	-91
2	3	44	54
-3	3	381	314
-4	3	200	-157
5	3	110	98
-5	3	297	314
-6	3	70	-41
7	3	72	39
-7	3	56	16
8	3	255	-241
-8	3	134	163
9	3	108	-184
-9	3	78	86
-10	3	189	-226
11	3	99	80
-11	3	197	169
-12	3	132	95
-13	3	241	-249
14	3	47	59
15	3	61	-50
-15	3	256	-236
16	3	84	-70
-16	3	41	33
-17	3	44	27
18	3	59	-48
-18	3	48	63
19	3	48	27
-21	3	84	76
22	3	88	131
-22	3	70	[illegible]
23	3	97	40
-23	3	70	100
0	4	260	278
1	4	159	176
2	4	53	93
-2	4	95	114
-3	4	95	-119
4	4	65	-90
5	4	219	248
-5	4	326	356
6	4	46	34
7	4	160	173
-7	4	98	112
-9	4	159	196
10	4	78	-78
-10	4	59	54
12	4	125	-139
-12	4	209	-295
-13	4	51	-61
14	4	82	82
-14	4	85	83
-16	4	65	91
18	4	39	-31
-18	4	70	84
22	4	57	-79
-23	4	49	75
0	5	90	-109
1	5	77	-62
-1	5	116	-112
-3	5	159	183
4	5	85	-93
-4	5	89	60
5	5	75	-65
-5	5	331	-281
-7	5	140	-142
8	5	88	89
-9	5	343	-344
-11	5	54	-26
-13	5	102	97
-15	5	178	187
-16	5	76	-32
-17	5	96	117
-19	5	52	72
-20	5	39	14
-21	5	56	-47
-22	5	52	90
-23	5	89	-122
2	6	278	-303
-2	6	112	103
3	6	44	18
-3	6	73	-84
4	6	82	-101
-4	6	124	-142
5	6	127	-129
-5	6	56	-60
6	6	186	183
-6	6	70	-116
7	6	65	-65
-7	6	79	-93
8	6	66	-58
-8	6	103	-112
9	6	114	114
-13	6	61	35
-15	6	64	77
18	6	39	-33
-19	6	46	-52
-20	6	122	-147
-21	6	59	58
-1	7	40	57
2	7	69	72
3	7	46	-43
-7	7	64	-84
8	7	107	-138
-9	7	113	115
11	7	80	-76
-11	7	42	39
13	7	42	-10
-14	7	56	42
15	7	60	-97
-15	7	167	-186
-17	7	87	-127
-18	7	101	117
-19	7	86	-98
1	8	44	-32
4	8	40	-48
5	8	43	-72
7	8	41	62
-7	8	42	23
9	8	79	-83
10	8	41	41
-10	8	39	44
11	8	96	-140
12	8	40	29
-12	8	101	-112
-13	8	99	-136
-14	8	53	-82
0	9	92	91
2	9	39	-21
-2	9	63	-74
3	9	65	65
-3	9	62	61
6	9	46	39
-12	9	40	-23
0	10	51	58
-2	10	58	61
••••••••••••••••			
K = 10			
0	0	428	416
2	0	167	207
4	0	143	-137
5	0	183	-183
8	0	319	-334
9	0	190	213
10	0	98	-74
11	0	165	-137
12	0	78	69
13	0	390	-363
14	0	136	-99
15	0	87	-62
16	0	99	-98
17	0	96	-72
18	0	126	-102
19	0	132	-113
20	0	86	-86
21	0	76	-92
23	0	64	63
0	1	148	-136
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5	4	42	-45
6	4	50	-85
-7	4	44	-53
-8	4	64	38
9	4	43	-31
-9	4	54	-87
10	4	54	27
-10	4	61	5
11	4	48	-10
13	4	54	43
1	5	90	79
2	5	56	-37
5	5	68	78
-5	5	78	114
7	5	50	-40
8	5	39	59
9	5	44	-49
11	5	40	91
12	5	47	-48
-13	5	43	-65
-14	5	56	80
0	6	81	-98
1	6	117	-96
3	6	66	-65
-3	6	86	-115
5	6	52	-20
-10	6	46	45
-3	7	40	-90
0	8	45	65
••••••••••••••••			
K = 16			
3	0	107	-82
5	0	85	-69
8	0	103	108
15	0	51	58
0	1	165	153
2	1	116	117
-3	1	104	99
-5	1	97	97
-7	1	75	69
8	1	63	-42
-10	1	62	-66
11	1	90	98
13	1	77	-54
-13	1	67	67
15	1	60	-50
16	1	64	85
17	1	94	-92
-17	1	99	82
18	1	44	77
-18	1	45	61
19	1	51	-55
-19	1	120	150
0	2	130	120
2	2	97	-105
3	2	146	108
-4	2	76	88
5	2	87	100
-7	2	88	-103
10	2	82	-98
-14	2	62	-73
-16	2	64	64
0	3	76	-85
-1	3	48	-56
2	3	78	-59
-3	3	81	81
4	3	61	47
5	3	66	-51
6	3	142	136
7	3	95	-90
-7	3	73	-68
8	3	130	147
-8	3	78	62
-9	3	41	-57
-17	3	84	-105
-2	4	39	20
-4	4	65	-94
-6	4	88	-121
10	4	53	-91
-11	4	52	83
12	4	55	-14
-12	4	59	-38
-15	4	61	-33
0	5	113	135
-3	5	106	-137
-4	5	42	9
7	5	49	-89
8	5	53	-83
9	5	64	81
-9	5	67	-95
0	6	40	30
3	6	66	72
-4	6	86	126
6	6	45	67
-7	6	45	65
2	7	75	-103
-2	7	71	-79
••••••••••••••••			
K = 17			
3	0	84	-88
8	0	87	-62
10	0	89	-88
12	0	91	74
14	0	95	95
16	0	65	58
17	0	62	-89
2	1	166	-142
-2	1	144	123
-6	1	64	73
-7	1	96	102
8	1	69	101
9	1	72	86
10	1	78	88
15	1	50	41
-15	1	46	54
16	1	72	77
-16	1	65	-48
-2	2	54	77
3	2	81	-81
4	2	103	99
5	2	91	-87
-5	2	80	104
6	2	88	98
7	2	64	-93
-9	2	68	-60
10	2	59	48
-12	2	52	40
-13	2	60	-50
14	2	46	-33
-14	2	55	68
15	2	81	-88
-16	2	50	35
-17	2	56	68
1	3	67	95
2	3	86	91
-6	3	46	-52
8	3	53	-55
-8	3	45	-50
-9	3	41	-58
-12	3	84	-96
-13	3	61	69
1	4	56	-57
4	4	43	-49
-6	4	49	85
5	4	42	92
6	4	53	-40
7	4	52	178
-7	4	52	78
-9	4	60	85
-12	4	48	18
2	5	76	-96
-5	5	67	-55
-6	5	41	51
-3	6	66	87
-5	6	63	45
••••••••••••••••			
K = 18			
0	0	129	-121
2	0	65	-69
4	0	64	57
5	0	40	55
6	0	74	82
7	0	72	61
10	0	64	69
12	0	55	-48
14	0	44	-39
15	0	50	-29
0	1	179	-178
-2	1	104	-96
3	1	80	-91
-3	1	58	41
4	1	83	-86
-4	1	110	-108
5	1	78	-91
6	1	115	-105
-6	1	78	-72
-11	1	115	-103
-13	1	67	-54
-14	1	51	52
-15	1	60	-77
1	2	84	-107
-1	2	97	98
6	2	68	-65
7	2	67	-44
-8	2	98	-100
9	2	51	-56
-9	2	42	44
11	2	49	45
12	2	42	27
0	3	99	113
1	3	65	79
3	3	64	73
4	3	56	-63
5	3	83	85
-5	3	52	-47
-7	3	58	-52
9	3	59	-71
-9	3	55	35
10	3	52	-74
-10	3	86	-99
-11	3	40	24
-12	3	49	-66
-13	3	67	62
-14	3	45	-63
0	4	57	53
4	4	55	29
-7	4	62	-99
0	5	39	-44
-1	6	68	44
-2	6	59	-74
••••••••••••••••			
K = 19			
5	0	139	161
7	0	53	50
1	2	57	-59
2	2	120	-129
-2	2	50	45
4	2	67	-67
5	2	40	-92
6	2	51	-77
7	2	46	-78
8	2	63	78
-3	3	39	-30
-6	3	51	-69
-8	3	50	46
-1	4	65	-46
2	4	77	85
-2	5	43	-54
••••••••••••••••			
K = 20			
2	0	58	-49
4	0	48	-37
6	0	49	-59
8	0	57	-67
9	0	59	67
-2	1	51	60
0	2	48	-52
-2	2	52	73
1	3	44	-20
-1	3	43	18
3	3	51	-53

[a] Tabulated by *h* index, F_o, and F_c. Unobserved reflections have been omitted from this list.

bols. Repeated application by hand of the relation

$$\phi_H \approx \langle \phi_K + \phi_{H-K} \rangle_K$$

where ϕ_H is the phase (0 or 180°) of reflection $H = hkl$ indicated the correct values of the symbols and determined 150 signs, of which 16 were erroneous. This phase set was corrected and extended by the use of Sayre's equation,[10] implemented by G. N. Reeke in the program FAZE. In 7 min of IBM 7094 time all 734 reflections having $|E| > 1.5$ were correctly phased. The E map revealed all B, N, and O atoms at heights of 918–1655 arbitrary units, eight C atoms at 447–739, and the highest false peak at 706. The atomic numbering scheme is shown in Figures 1–4. An electron density map with phases calculated from these atomic positions showed methyl carbons at heights 1.5–2.1 e⁻/Å³ and indicated disorder around N(37). The twofold disordered methylene carbons were located on a second electron density map at 1.1–1.4 e⁻/Å³ except for C(42) at only 0.7 e⁻/Å³. The CRYRM computing system[11] as

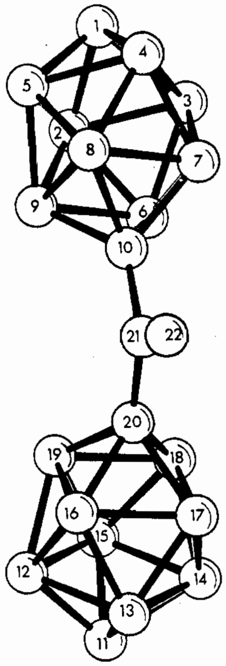

Figure 1.—Numbering system for the $B_{20}H_{18}NO^{3-}$ ion.

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[10] D. Sayre, *Acta Crystallogr.*, **5**, 60 (1952).

[11] D. J. Duchamp, Program and Abstracts, American Crystallographic Association Meeting, Bozeman, Mont., 1964, paper B-14.

TABLE II

POSITIONS AND ANISOTROPIC TEMPERATURE FACTORS[a] WITH STANDARD DEVIATIONS FOR B, C, N, AND O ATOMS

Atom	Pop.	x	y	z	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
B(1)	1.000	0.2445 (3)	0.1308 (6)	0.5281 (9)	12 (2)	37 (5)	89 (16)	2 (5)	4 (9)	14 (14)
B(2)	1.000	0.2042 (4)	0.2136 (6)	0.5499 (9)	15 (3)	41 (5)	81 (16)	−1 (6)	12 (10)	18 (15)
B(3)	1.000	0.1759 (4)	0.1200 (6)	0.4568 (10)	17 (3)	46 (6)	83 (17)	1 (6)	19 (10)	19 (16)
B(4)	1.000	0.2099 (4)	0.0562 (6)	0.6028 (10)	14 (3)	36 (5)	95 (16)	2 (6)	9 (10)	10 (15)
B(5)	1.000	0.2357 (4)	0.1484 (6)	0.6988 (10)	16 (2)	30 (5)	94 (16)	2 (6)	5 (9)	15 (15)
B(6)	1.000	0.1312 (4)	0.1882 (6)	0.5409 (10)	13 (2)	35 (5)	105 (16)	2 (6)	9 (10)	10 (15)
B(7)	1.000	0.1361 (4)	0.0744 (5)	0.5814 (9)	14 (2)	29 (5)	81 (15)	2 (5)	13 (9)	2 (14)
B(8)	1.000	0.1776 (4)	0.0945 (6)	0.7476 (9)	15 (2)	33 (5)	82 (15)	2 (6)	5 (9)	19 (14)
B(9)	1.000	0.1731 (4)	0.2075 (6)	0.7115 (9)	14 (2)	30 (5)	89 (15)	−4 (6)	3 (9)	11 (14)
B(10)	1.000	0.1169 (4)	0.1444 (5)	0.6953 (9)	14 (2)	27 (5)	74 (15)	2 (5)	−3 (9)	14 (13)
B(11)	1.000	−0.0697 (4)	0.3881 (6)	0.7446 (4)	17 (3)	37 (6)	117 (17)	−7 (6)	26 (10)	−24 (16)
B(12)	1.000	−0.0110 (4)	0.3685 (6)	0.8571 (10)	15 (2)	35 (5)	92 (16)	2 (6)	16 (10)	10 (15)
B(13)	1.000	−0.0710 (4)	0.2989 (6)	0.8298 (10)	15 (3)	33 (6)	110 (16)	0 (6)	18 (10)	−10 (15)
B(14)	1.000	−0.0727 (4)	0.3040 (6)	0.6417 (9)	14 (2)	32 (5)	95 (16)	4 (6)	9 (9)	−14 (15)
B(15)	1.000	−0.0125 (4)	0.3755 (6)	0.6650 (10)	13 (2)	35 (5)	99 (16)	3 (6)	8 (10)	−15 (15)
B(16)	1.000	−0.0032 (4)	0.2588 (5)	0.8874 (9)	14 (2)	31 (5)	74 (14)	4 (5)	15 (9)	−15 (13)
B(17)	1.000	−0.0453 (4)	0.2151 (6)	0.7363 (10)	13 (2)	31 (5)	106 (15)	5 (6)	17 (9)	−15 (15)
B(18)	1.000	−0.0023 (3)	0.2708 (5)	0.6145 (9)	11 (2)	33 (5)	74 (14)	2 (5)	4 (8)	−2 (13)
B(19)	1.000	0.0400 (4)	0.3150 (5)	0.7656 (9)	11 (2)	28 (5)	92 (14)	0 (5)	8 (9)	4 (14)
B(20)	1.000	0.0257 (3)	0.2146 (5)	0.7544 (9)	12 (2)	24 (5)	78 (14)	0 (5)	16 (8)	9 (13)
N(21)	1.000	0.0640 (2)	0.1472 (4)	0.7609 (6)	16 (1)	29 (3)	83 (8)	−6 (3)	5 (5)	2 (8)
O(22)	1.000	0.0515 (2)	0.0839 (3)	0.8245 (6)	21 (1)	22 (2)	165 (8)	2 (3)	41 (5)	49 (7)
N(23)	1.000	0.2372 (3)	0.2070 (5)	0.1987 (7)	21 (2)	72 (4)	81 (9)	−12 (4)	13 (6)	11 (11)
C(24)	1.000	0.2781 (5)	0.1384 (9)	0.1452 (13)	41 (4)	106 (9)	187 (20)	14 (9)	79 (13)	25 (22)
C(25)	1.000	0.2700 (4)	0.2821 (7)	0.2279 (11)	32 (3)	66 (6)	156 (16)	−19 (7)	2 (10)	14 (16)
C(26)	1.000	0.1852 (5)	0.2096 (9)	0.1057 (11)	38 (3)	134 (9)	121 (16)	−58 (9)	−16 (11)	108 (19)
C(27)	1.000	0.2612 (5)	0.0614 (9)	0.1551 (12)	29 (3)	103 (8)	191 (19)	30 (8)	7 (11)	−57 (21)
C(28)	1.000	0.2867 (5)	0.3250 (7)	0.1001 (13)	35 (3)	70 (7)	278 (21)	−4 (7)	77 (13)	117 (19)
C(29)	1.000	0.1418 (4)	0.2633 (7)	0.1586 (12)	34 (3)	93 (7)	204 (17)	54 (7)	44 (11)	131 (17)
N(30)	1.000	0.4151 (3)	0.4177 (4)	0.6822 (7)	20 (2)	25 (3)	170 (11)	−1 (4)	1 (6)	−25 (10)
C(31)	1.000	0.3620 (5)	0.3857 (7)	0.7261 (14)	43 (3)	45 (6)	301 (24)	−16 (7)	59 (14)	−59 (19)
C(32)	1.000	0.4250 (5)	0.4099 (7)	0.5338 (14)	45 (3)	45 (6)	252 (21)	11 (7)	32 (13)	−57 (18)
C(33)	1.000	0.4637 (7)	0.3805 (8)	0.7727 (18)	49 (4)	49 (7)	488 (35)	30 (9)	−86 (20)	−41 (26)
C(34)	1.000	0.3527 (6)	0.2977 (9)	0.7065 (17)	57 (5)	75 (9)	403 (33)	−64 (10)	60 (19)	−34 (28)
C(35)	1.000	0.3861 (6)	0.4556 (8)	0.4388 (12)	57 (4)	103 (8)	157 (18)	63 (9)	−8 (13)	−126 (19)
C(36)	1.000	0.5100 (6)	0.4171 (10)	0.8012 (20)	44 (4)	81 (9)	653 (43)	0 (10)	−180 (23)	36 (33)
N(37)	1.000	0.4018 (3)	0.0049 (4)	0.7433 (7)	17 (2)	39 (3)	132 (10)	7 (4)	4 (6)	−20 (10)
C(38)	0.562	0.3507 (8)	−0.0250 (12)	0.8034 (22)	28 (5)	59 (10)	224 (33)	17 (11)	47 (20)	−13 (30)
C(39)	0.562	0.4290 (7)	0.0777 (10)	0.8165 (18)	27 (4)	32 (7)	149 (26)	−12 (9)	6 (17)	−16 (23)
C(40)	0.562	0.3863 (7)	0.0303 (11)	0.5843 (17)	29 (4)	45 (9)	112 (23)	5 (10)	−36 (16)	15 (23)
C(41)	0.438	0.3886 (9)	0.0079 (14)	0.9133 (25)	25 (6)	49 (11)	191 (37)	21 (12)	−5 (23)	−57 (32)
C(42)	0.438	0.4297 (14)	0.0832 (28)	0.7120 (39)	45 (9)	203 (35)	238 (69)	−84 (29)	45 (41)	−20 (83)
C(43)	0.438	0.3499 (8)	−0.0187 (13)	0.6653 (26)	13 (4)	51 (12)	243 (39)	33 (11)	−24 (21)	−22 (34)
C(44)	1.000	0.3686 (4)	−0.0618 (7)	0.9569 (11)	33 (3)	59 (6)	180 (17)	−3 (6)	70 (11)	−32 (16)
C(45)	1.000	0.4804 (6)	0.1023 (7)	0.7558 (12)	50 (4)	65 (7)	171 (18)	−36 (8)	19 (14)	−51 (18)
C(46)	1.000	0.3616 (4)	−0.0345 (7)	0.5034 (10)	29 (3)	65 (6)	139 (15)	9 (6)	−27 (10)	−9 (15)

[a] The x, y, and z are in fractional monoclinic coordinates. Temperature coefficients occur in the expression $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})]$.

modified by G. N. Reeke was used in all electron density, bond distance, and least-squares calculations. Least-squares refinements were carried out with the use of atomic scattering factors from ref 12 to minimize the function $\Sigma w(|F_o|^2 - |F_c|^2)^2$ in which w was taken as $1/F_o^2$ for $F_o \geq 12.8$ and $1/(12.8F_o)$ for $F_o < 12.8$. Unobserved structure factors were included as $F_{\min}$ only if $F_c > F_{\min}$. The first isotropic refinement, on the small data set, with atom C(42) omitted, began at discrepancy indices $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.28$ and $R_w = [\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w|F_o|^4]^{1/2} = 0.50$, proceeded to $R = 0.19$ and $R_w = 0.30$, but yielded an unreasonably high temperature factor for C(39). Introduction of C(42) and refinement of population factors for the two sets of methylene carbon positions with temperature factors held equal resulted in average occupancies of 0.56 and 0.44. Further isotropic refinement lowered R to 0.15 and R_w to 0.30. Hydrogens attached to boron were clearly visible on a difference electron density map at 0.2–0.4 e$^-$/Å^3, but triethylammonium hydrogens were not resolved and had to be introduced at calculated positions. The numbering system identifies cage hydrogens by the number of the attached boron plus 60. Triethylammonium hydrogens are numbered sequentially by N or C atom, with H(81) bonded to N(23), H(82) and H(83) bonded to C(24), etc. Isotropic refinements, in which hydrogens were given the isotropic temperature factor of the attached atom and not refined, converged to $R = 0.146$ and $R_w = 0.292$. With the larger set of 4555 reflections the same parameters

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(12) "International Tables for X-Ray Crystallography," Vol. 3, Kynoch Press, Birmingham, England, 1962, pp 202–203.

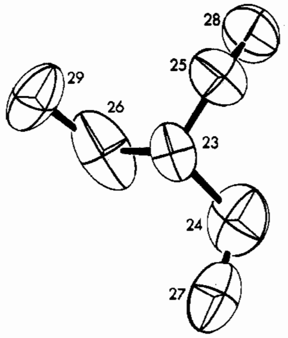

Figure 2.—Numbering system and thermal ellipsoids for the first ordered triethylammonium ion.

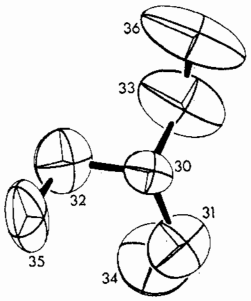

Figure 3.—Numbering system and thermal ellipsoids for the second ordered triethylammonium ion.

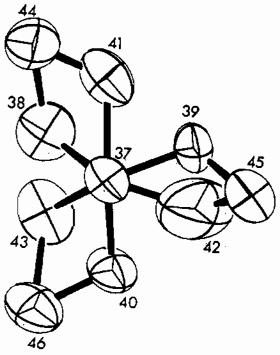

Figure 4.—Numbering system and thermal elliposids for the disordered triethylammonium ion.

yielded $R = 0.187$ and $R_w = 0.385$. Separate least-squares refinements of anisotropic temperature factors and coordinates of nonhydrogen atoms with a separate matrix block for each ion and a shift factor of $^2/_3$, followed by adjustment of hydrogen positions lying on gradients in a difference electron density map, produced final values $R = 0.141$ and $R_w = 0.240$. The refinement had not fully converged since calculated shifts in the last cycle were still as much as σ for coordinates and 5σ for temperature factors.

To avoid bias the NO group was refined as two nitrogen atoms until the final anisotropic refinement. The isotropic temperature factors provided evidence that atom 21 ($B = 3.43$ Å^2) is nitrogen and 22 ($B = 2.75$ Å^2) is oxygen since the more movable atom 22 should have the higher B. Supporting evidence is furnished by electron density maps in the plane 10–20–21–22 and in the perpendicular plane through 21–22. If atom 22 were N, one would expect it to bear an attached hydrogen, but the largest peak 1.1 Å from atom 22 not interfering with atom 21 is only 0.11 e$^-$/Å^3 in height (F_{000} included).

TABLE III

POSITIONS[a] AND ASSIGNED ISOTROPIC TEMPERATURE FACTORS[b] FOR CAGE HYDROGENS

Atom	x	y	z	β
H(61)	0.288 (6)	0.135 (9)	0.477 (15)	3.33
H(62)	0.215 (6)	0.283 (9)	0.528 (15)	3.64
H(63)	0.168 (6)	0.106 (9)	0.346 (15)	3.93
H(64)	0.229 (6)	−0.004 (9)	0.615 (15)	2.94
H(65)	0.276 (6)	0.154 (9)	0.785 (15)	3.21
H(66)	0.098 (6)	0.222 (9)	0.459 (15)	3.41
H(67)	0.109 (6)	0.022 (9)	0.529 (15)	2.96
H(68)	0.182 (6)	0.061 (9)	0.855 (15)	3.46
H(69)	0.177 (6)	0.263 (9)	0.779 (15)	3.18
H(71)	−0.098 (6)	0.444 (9)	0.749 (15)	3.63
H(72)	0.005 (6)	0.419 (9)	0.937 (15)	3.33
H(73)	−0.107 (6)	0.289 (9)	0.893 (15)	3.58
H(74)	−0.109 (6)	0.292 (9)	0.539 (15)	3.20
H(75)	0.003 (6)	0.430 (9)	0.597 (15)	3.17
H(76)	0.008 (6)	0.228 (9)	0.997 (15)	2.59
H(77)	−0.066 (6)	0.157 (9)	0.709 (15)	3.17
H(78)	0.005 (6)	0.257 (9)	0.498 (15)	2.53
H(79)	0.087 (6)	0.329 (9)	0.785 (15)	2.62

[a] With estimated errors. [b] Set equal to the final refined isotropic temperature factor of the attached boron.

In view of the short bond distances C(24)–C(27) and C(33)–C(36) and the methylene disorder, a difference electron density map was made with the offending ethyl groups omitted from the calculation. The atoms all returned at their previous sites, and no new peaks appeared in chemically reasonable positions. The last difference electron density map, made at the $R = 0.15$, $R_w = 0.26$ stage with C(42) omitted, displayed C(42) at 0.7 e$^-$/Å^3, atomic residuals to 0.5 e$^-$/Å^3, negative regions within the boron cages of −0.5 e$^-$/Å^3, and noise peaks to 0.4 e$^-$/Å^3.

Results and Discussion

Positions and temperature factors are presented in Table II for anisotropically refined atoms, in Table III for borane terminal hydrogens, and in Table IV as assigned to triethylammonium hydrogens. Thermal motion is analyzed for direction of principal axes and rms amplitude in Table V. Bond distances, bond angles, and shortest nonbonded interactions are listed in Tables VI–VIII. Drawings of the ions, in which the thermal ellipsoids correspond to 50% probability, were prepared with C. K. Johnson's program ORTEP and appear in Figures 1–5.

The $B_{20}H_{18}NO^{3-}$ ion is closely related to a model of C_{2v} symmetry in which the twofold axis passes through N(21)–O(22), BNB and BNO angles are 120°, and a symmetry plane containing B(10)–B(20)–N(21)–O(22) relates atoms B(8)–B(9)–B(16)–B(19) in a parallel plane above to B(7)–B(6)–B(17)–B(18) in the parallel plane below. This symmetrical model has closest H–H contacts of 2.3 Å between opposite cages. Contacts this short are prevented in the actual structure by rotating the first cage 10.5° counterclockwise about the 1–10

TABLE IV

CALCULATED POSITIONS[a] AND ASSIGNED TEMPERATURE FACTORS[b] FOR TRIETHYLAMMONIUM HYDROGENS

Atom	Pop.	x	y	z	β
81[c]	1.000	0.223 (6)	0.207 (9)	0.304 (15)	4.66
82	1.000	0.320 (8)	0.142 (12)	0.205 (21)	9.67
83	1.000	0.284 (8)	0.148 (12)	0.035 (21)	9.67
84	1.000	0.307 (8)	0.270 (12)	0.303 (21)	7.14
85	1.000	0.244 (8)	0.325 (12)	0.287 (21)	7.14
86	1.000	0.168 (8)	0.147 (12)	0.092 (21)	8.48
87	1.000	0.194 (8)	0.231 (12)	0.000 (21)	8.48
88	1.000	0.288 (8)	0.011 (12)	0.121 (21)	8.49
89	1.000	0.256 (8)	0.044 (12)	0.267 (21)	8.49
90	1.000	0.220 (8)	0.050 (12)	0.097 (21)	8.49
91	1.000	0.312 (8)	0.288 (12)	0.043 (21)	8.01
92	1.000	0.309 (8)	0.385 (12)	0.122 (21)	8.01
93	1.000	0.248 (8)	0.343 (12)	0.028 (21)	8.01
94	1.000	0.132 (8)	0.242 (12)	0.260 (21)	7.50
95	1.000	0.158 (8)	0.326 (12)	0.167 (21)	7.50
96	1.000	0.103 (8)	0.266 (12)	0.087 (21)	7.50
97[c]	1.000	0.421 (6)	0.482 (9)	0.706 (15)	4.27
98	1.000	0.358 (8)	0.402 (12)	0.833 (21)	9.22
99	1.000	0.326 (8)	0.419 (12)	0.660 (21)	9.22
100	1.000	0.466 (8)	0.430 (12)	0.522 (21)	7.94
101	1.000	0.420 (8)	0.345 (12)	0.507 (21)	7.94
102[c]	1.000	0.442 (8)	0.370 (12)	0.875 (21)	12.46
103	1.000	0.473 (8)	0.322 (12)	0.721 (21)	12.46
104	1.000	0.313 (8)	0.274 (12)	0.733 (21)	13.08
105	1.000	0.386 (8)	0.265 (12)	0.767 (21)	13.08
106	1.000	0.354 (8)	0.282 (12)	0.594 (21)	13.08
107	1.000	0.343 (8)	0.434 (12)	0.451 (21)	9.50
108	1.000	0.391 (8)	0.448 (12)	0.330 (21)	9.50
109	1.000	0.389 (8)	0.519 (12)	0.466 (21)	9.50
110	1.000	0.543 (8)	0.390 (12)	0.873 (21)	13.42
111	1.000	0.525 (8)	0.441 (12)	0.713 (21)	13.42
112[c]	1.000	0.496 (8)	0.459 (12)	0.873 (21)	13.42
113[c]	1.000	0.436 (6)	−0.047 (9)	0.760 (15)	4.19
114	0.562	0.333 (8)	−0.078 (12)	0.725 (21)	6.62
115[c]	0.562	0.323 (8)	0.025 (12)	0.804 (21)	6.62
116	0.562	0.440 (8)	0.064 (12)	0.931 (21)	5.27
117	0.562	0.400 (8)	0.129 (12)	0.805 (21)	5.27
118	0.562	0.423 (8)	0.044 (12)	0.536 (21)	5.39
119	0.562	0.357 (8)	0.078 (12)	0.574 (21)	5.39
120	0.438	0.425 (8)	0.023 (12)	0.975 (21)	5.62
121	0.438	0.357 (8)	0.054 (12)	0.916 (21)	5.62
122[c]	0.438	0.420 (8)	0.146 (12)	0.731 (21)	11.62
123	0.438	0.417 (8)	0.093 (12)	0.601 (21)	11.62
124	0.438	0.335 (8)	−0.076 (12)	0.712 (21)	5.73
125	0.438	0.320 (8)	0.032 (12)	0.670 (21)	5.73
126	0.562	0.330 (8)	−0.085 (12)	0.988 (21)	6.68
127	0.562	0.396 (8)	−0.114 (12)	0.947 (21)	6.68
128	0.562	0.383 (8)	−0.012 (12)	1.022 (21)	6.68
129	0.438	0.359 (8)	−0.061 (12)	1.062 (21)	6.68
130	0.438	0.398 (8)	−0.111 (12)	0.945 (21)	6.68
131	0.438	0.330 (8)	−0.080 (12)	0.886 (21)	6.68
132	0.562	0.501 (8)	0.154 (12)	0.811 (21)	8.32
133	0.562	0.511 (8)	0.051 (12)	0.773 (21)	8.32
134	0.562	0.471 (8)	0.116 (12)	0.646 (21)	8.32
135	0.438	0.509 (8)	0.053 (12)	0.725 (21)	8.32
136	0.438	0.500 (8)	0.159 (12)	0.735 (21)	8.32
137	0.438	0.491 (8)	0.089 (12)	0.870 (21)	8.32
138	0.562	0.322 (8)	−0.053 (12)	0.546 (21)	6.16
139	0.562	0.388 (8)	−0.087 (12)	0.508 (21)	6.16
140	0.562	0.349 (8)	−0.020 (12)	0.392 (21)	6.16
141	0.438	0.320 (8)	−0.052 (12)	0.446 (21)	6.16
142	0.438	0.388 (8)	−0.087 (12)	0.500 (21)	6.16
143	0.438	0.373 (8)	0.021 (12)	0.458 (21)	6.16

[a] Calculated after all carbon and nitrogen atoms had been located by setting the C–H or N–H distance to 1.095 Å and making all bonded H–C–C and H–C–N angles and two of three bonded H–N–C angles exactly tetrahedral, with methyl hydrogens staggered with respect to methylene hydrogens. Errors are guessed to be generally 0.2 Å along each axis. [b] Assigned equal to the isotropic temperature factor of the atom to which the hydrogen atom is bonded. [c] Calculated positions slightly modified as indicated by difference Fourier maps.

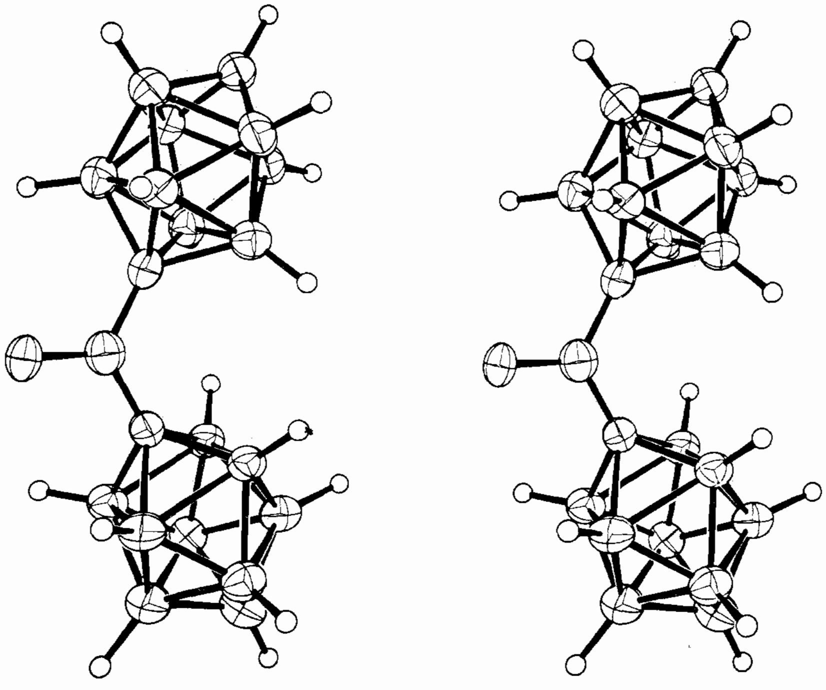

Figure 5.—Stereoviews with thermal ellipsoids of the $B_{20}H_{18}NO^{3-}$ ion.

TABLE V: RMS AMPLITUDES AND DIRECTION COSINES[a] OF THERMAL MOTION

Atom	Rms amp, Å	Cos *A*	Cos *B*	Cos *C*	Atom	Rms amp, Å	Cos *A*	Cos *B*	Cos *C*
B(1)	0.234	0.018	0.920	0.387	C(24)	0.402	0.492	0.799	0.285
	0.202	−0.497	−0.332	0.856		0.357	0.664	−0.602	0.359
	0.182	0.867	−0.210	0.342		0.249	−0.563	−0.012	0.889
B(2)	0.246	−0.099	0.953	0.297	C(25)	0.344	−0.737	0.644	0.294
	0.212	0.955	0.000	0.178		0.275	−0.624	−0.765	0.233
	0.186	−0.280	−0.303	0.938		0.265	0.258	−0.012	0.927
B(3)	0.258	0.135	0.946	0.275	C(26)	0.490	−0.486	0.839	0.302
	0.227	0.920	−0.230	0.203		0.284	0.871	0.443	0.103
	0.185	−0.368	−0.227	0.940		0.207	−0.069	−0.315	0.948
B(4)	0.229	0.178	0.891	0.393	C(27)	0.410	−0.391	−0.874	0.333
	0.209	−0.693	−0.188	0.775		0.287	0.371	0.136	0.866
	0.203	0.698	−0.414	0.494		0.267	−0.842	0.466	0.372
B(5)	0.224	−0.612	0.436	0.729	C(28)	0.411	0.264	0.517	0.776
	0.218	0.763	0.545	0.252		0.326	−0.825	0.558	0.014
	0.191	0.207	−0.716	0.636		0.222	−0.500	−0.649	0.631
B(6)	0.229	0.022	0.709	0.697	C(29)	0.440	0.458	0.754	0.411
	0.212	−0.241	−0.681	0.716		0.287	−0.761	0.060	0.734
	0.199	0.970	−0.185	0.035		0.219	0.459	−0.654	0.541
B(7)	0.212	0.778	0.516	0.261	N(30)	0.291	−0.364	−0.194	0.949
	0.200	0.442	−0.855	0.215		0.241	0.930	−0.130	0.228
	0.190	−0.447	0.052	0.941		0.182	0.052	0.972	0.220
B(8)	0.227	0.051	0.867	0.487	C(31)	0.398	0.508	−0.275	0.748
	0.216	−0.983	−0.045	0.299		0.335	−0.852	−0.021	0.624
	0.182	0.178	−0.497	0.821		0.236	0.126	0.961	0.227
B(9)	0.225	−0.582	0.623	0.590	C(32)	0.367	−0.981	−0.195	0.139
	0.197	0.681	0.023	0.643		0.354	0.084	−0.329	0.923
	0.191	−0.444	−0.782	0.489		0.228	−0.177	0.924	0.358
B(10)	0.215	−0.813	0.258	0.618	C(33)	0.533	−0.565	−0.149	0.874
	0.203	0.458	0.838	0.240		0.341	0.749	0.320	0.484
	0.169	0.361	−0.481	0.749		0.243	0.346	−0.936	0.026
B(11)	0.262	0.450	−0.585	0.615	C(34)	0.466	0.713	−0.475	0.425
	0.212	0.443	0.802	0.343		0.420	−0.456	0.245	0.905
	0.204	−0.776	0.119	0.710		0.256	−0.533	−0.845	0.024
B(12)	0.230	0.345	0.815	0.419	C(35)	0.493	−0.716	−0.650	0.339
	0.210	0.674	−0.560	0.396		0.325	0.664	−0.524	0.448
	0.195	−0.653	−0.147	0.817		0.204	−0.214	0.550	0.827
B(13)	0.234	0.305	−0.438	0,802	C(36)	0.628	−0.530	0.049	0.905
	0.214	0.441	0.852	0.227		0.339	0.145	0.989	0.015
	0.202	−0.844	0.286	0.553		0.269	0.836	−0.140	0.425
B(14)	0.229	−0.414	−0.745	0.569	N(37)	0.266	−0.421	−0.571	0.751
	0.208	0.710	0.094	0.605		0.228	0.495	0.507	0.640
	0.193	−0.569	0.660	0.556		0.208	−0.760	0.646	0.162
B(15)	0.235	−0.243	−0.805	0.568	C(38)	0.334	0.483	0.146	0.798
	0.205	0.327	0.458	0.780		0.310	−0.477	−0.783	0.454
	0.195	−0.913	0.378	0.262		0.241	−0.735	0.604	0.396
B(16)	0.217	−0.378	−0.887	0.311	C(39)	0.292	−0.947	0.249	0.319
	0.205	0.800	−0.167	0.475		0.266	0.128	−0.292	0.925
	0.167	−0.467	0.431	0.824		0.197	0.296	0.923	0.207
B(17)	0.231	−0.006	−0.549	0.830	C(40)	0.327	−0.894	−0.040	0.552
	0.215	0.656	0.629	0.335		0.257	0.082	0.965	0.238
	0.179	−0.755	0.550	0.446		0.189	0.441	−0.260	0.799
B(18)	0.215	−0.155	−0.983	0.120	C(41)	0.346	−0.539	−0.541	0.707
	0.186	−0.424	0.159	0.937		0.264	0.681	0.172	0.623
	0.176	0.893	−0.095	0.328		0.215	−0.496	0.823	0.334
B(19)	0.208	−0.084	0.382	0.924	C(42)	0.569	0.407	−0.912	0.008
	0.196	0.093	−0.917	0.374		0.337	0.338	0.210	0.869
	0.185	−0.992	−0.118	0.080		0.299	−0.848	−0.354	0.495
B(20)	0.204	0.564	0.348	0.674	C(43)	0.356	−0.409	−0.269	0.915
	0.185	−0.636	0.762	0.201		0.295	0.369	0.825	0.380
	0.172	−0.527	−0.546	0.711		0.124	0.835	−0.497	0.134
N(21)	0.225	−0.851	0.503	0.253	C(44)	0.349	0.705	−0.260	0.568
	0.196	0.000	−0.287	0.951		0.289	−0.382	−0.923	0.091
	0.192	0.525	0.815	0.179		0.233	−0.598	0.283	0.818
O(22)	0.300	0.396	0.292	0.816	C(45)	0.408	0.888	−0.444	0.009
	0.228	−0.901	0.304	0.416		0.309	−0.384	−0.575	0.764
	0.152	−0.174	−0.907	0.402		0.243	0.252	0.688	0.645
N(23)	0.325	−0.280	0.959	0.084	C(46)	0.336	−0.781	−0.461	0.514
	0.241	0.943	0.265	0.085		0.294	−0.381	0.886	0.307
	0.191	−0.181	−0.103	0.993		0.226	0.494	−0.044	0.801

[a] Direction cosines of principal axes are given relative to the crystallographic axes.

TABLE VI
BOND DISTANCES [Å][a]

	N–O		
21–22	1.28 (2)		
	Cage–N		
10–21	1.51 (3)	20–21	1.46 (3)
	Cage Apex–Edge		
1–2	1.72 (4)	11–12	1.72 (4)
1–3	1.74 (4)	11–13	1.70 (4)
1–4	1.71 (4)	11–14	1.71 (4)
1–5	1.71 (4)	11–15	1.69 (4)
6–10	1.73 (4)	16–20	1.71 (4)
7–10	1.71 (4)	17–20	1.72 (4)
8–10	1.72 (4)	18–20	1.72 (4)
9–10	1.72 (4)	19–20	1.71 (4)
	Cage Basal Edge–Edge		
2–3	1.89 (4)	12–13	1.86 (4)
3–4	1.88 (4)	13–14	1.81 (4)
4–5	1.86 (4)	14–15	1.89 (4)
2–5	1.89 (4)	12–15	1.85 (4)
6–7	1.94 (4)	16–17	1.83 (4)
7–8	1.82 (4)	17–18	1.91 (4)
8–9	1.92 (4)	18–19	1.84 (4)
6–9	1.86 (4)	16–19	1.92 (4)
	Cage Belt Edge–Edge		
2–6	1.83 (4)	12–16	1.86 (4)
2–9	1.82 (4)	12–19	1.84 (4)
3–6	1.83 (4)	13–16	1.81 (4)
3–7	1.80 (4)	13–17	1.82 (4)
4–7	1.82 (4)	14–17	1.82 (4)
4–8	1.80 (4)	14–18	1.86 (4)
5–8	1.79 (4)	15–18	1.84 (4)
5–9	1.84 (4)	15–19	1.82 (4)
	Terminal B–H		
1–61	1.2 (1)	11–71	1.2 (1)
2–62	1.2 (1)	12–72	1.2 (1)
3–63	1.1 (1)	13–73	1.1 (1)
4–64	1.1 (1)	14–74	1.3 (1)
5–65	1.2 (1)	15–75	1.2 (1)
6–66	1.2 (1)	16–76	1.2 (1)
7–67	1.2 (1)	17–77	1.1 (1)
8–68	1.2 (1)	18–78	1.2 (1)
9–69	1.1 (1)	19–79	1.2 (1)
	Nitrogen–Methylene Carbon		
23–24	1.64 (5)	30–31	1.51 (4)
23–25	1.49 (4)	30–32	1.49 (4)
23–26	1.46 (4)	30–33	1.52 (5)
37–38	1.52 (6)	37–41	1.71 (7)
37–39	1.52 (5)	37–42	1.52 (13)
37–40	1.59 (5)	37–43	1.45 (7)
	Methylene–Methyl Carbon		
24–27	1.36 (6)	31–34	1.49 (6)
25–28	1.52 (5)	32–35	1.45 (5)
26–29	1.52 (5)	30–36	1.29 (7)
38–44	1.61 (7)	41–44	1.35 (8)
39–45	1.51 (7)	42–45	1.30 (12)
40–46	1.42 (6)	43–46	1.64 (8)

[a] Errors have been propagated from standard deviations obtained in refinement of positions only, ignoring interaction with thermal parameters, and have been multiplied by a safety factor of 3.

axis (viewed from 1 to 10) and the second cage 5.5° counterclockwise about 11–20, thereby allowing the hydrogens to interleave, and by opening the BNB angle to 126°. Even so, two hydrogens (presumably 66 and 79) cannot be replaced by chlorine and four (66, 69, 78, and 79) cannot be replaced by bromine under forcing conditions[4] evidently because of close contacts: 66–78, 2.4; 66–79, 3.7; 69–78, 4.7; 69–79, 2.5 (2) Å. Atoms 10, 20, 21, and 22 still deviate by less than 0.01 Å from an unweighted least-squares plane. The B–N distances of 1.51 (3) and 1.46 (3) Å are intermediate between the accepted B–N single bond distance[13] of 1.58 and the shortened value[14] of 1.44 (2) Å in borazine. The long N–O distance of 1.28 (2) Å compares most closely with three-electron bond distances of 1.28 and 1.23 Å in the radicals di-*tert*-butyl nitroxide[15] and di-*p*-anisyl nitroxide.[16] These unusual bond distances in the nonradical species $B_{20}H_{18}NO^{3-}$ appear to be the result of electron delocalization to create a weaker than double N–O bond and stronger than single B–N bonds and thereby to improve the charge distribution as well. Within statistical significance the B–B distances are unchanged from those[17] in $Cu_2B_{10}H_{10}$. Averaged by type of interaction and listed in order for the 1–10 cage, the 11–20 cage, and $Cu_2B_{10}H_{10}$, they are as follows: apex–edge, 1.72 (1), 1.71 (2), 1.73 (2); basal edge–edge, 1.88 (4), 1.86 (4), 1.86 (3); belt edge–edge, 1.81 (2), 1.84 (2), 1.82 (2) Å.

There is evidence for hydrogen bonding in the short distance between O(22s) (transformation by the screw axis is indicated by s) and ammonium hydrogen 97 of 1.9 (2) Å, the angle 21s–22s–97 at the oxygen of 123 (7)°, in the angle 22s–97–30 at the hydrogen of 146 (10)°, and possibly in the long N(21)–O(22) bond distance. The position of H(97) should be reliable since its coordinates were refined with the aid of a difference electron density map. Thermal parameters (Table V) also suggest hydrogen bonding. While thermal motion of the boron atoms and N(21) is relatively small and isotropic, as expected for such a rigid framework, O(22) shows significant anisotropy. The largest rms vibration amplitude is 0.30 Å along principal axis 1, which makes angles of 87 and 70° to the O–N and O···H bonds. The amplitude is 0.23 Å along axis 2 at an 80° angle to O–N but closer (50°) to O···H and only 0.15 Å along axis 3 at angles 37 and 47°. Likewise, the amplitude of 0.18 Å along principal axis 3 of N(30) is the lowest in any triethylammonium group, and this axis lies roughly along the N–H bond.

As discussed in an earlier communication,[18] the disordered triethylammonium group centered at N(37) has a flat "pinwheel" conformation of approximate C_3 symmetry. The other two ordered triethylammonium ions differ by rotation of one ethyl group each, C(25)–C(28) and C(31)–C(34), through approximately 120° about the C–N bond to another staggered conformation. Bond distances are seriously affected by thermal motion

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TABLE VII

BOND ANGLES (DEG)

Across N–O Bridge

10–21–22	116.4 (1.7)	20–21–22	117.8 (1.5)
10–21–20	125.8 (1.9)		

Boron Cage Framework Angles

At Apices

2–1–3	66.0 (1.6)	12–11–13	66.0 (1.8)
3–1–4	65.7 (1.6)	13–11–14	64.1 (1.7)
4–1–5	65.9 (1.7)	14–11–15	67.3 (1.8)
5–1–2	66.7 (1.6)	15–11–12	65.8 (1.8)
6–10–7	68.6 (1.6)	16–20–17	64.6 (1.6)
7–10–8	64.1 (1.6)	17–20–18	67.5 (1.5)
8–10–9	67.6 (1.8)	18–20–19	64.8 (1.5)
9–10–6	65.0 (1.6)	19–20–16	68.3 (1.6)

At Equatorial Atoms, Neither Leg across Equatorial Belt

1–2–3	57.5 (1.5)	11–12–13	56.5 (1.6)
1–2–5	56.4 (1.5)	11–12–15	56.3 (1.6)
1–3–4	56.4 (1.5)	11–13–14	58.3 (1.7)
1–3–2	56.5 (1.6)	11–13–12	57.5 (1.6)
1–4–5	57.0 (1.6)	11–14–15	55.7 (1.6)
1–4–3	57.9 (1.5)	11–14–13	57.6 (1.6)
1–5–2	57.0 (1.5)	11–15–12	57.9 (1.7)
1–5–4	57.0 (1.6)	11–15–14	56.9 (1.6)
10–6–7	55.1 (1.4)	20–16–17	58.1 (1.5)
10–6–9	57.2 (1.5)	20–16–19	56.0 (1.4)
10–7–8	58.4 (1.6)	20–17–18	56.1 (1.4)
10–7–6	56.4 (1.4)	20–17–16	57.3 (1.5)
10–8–9	56.2 (1.5)	20–18–19	57.5 (1.4)
10–8–7	57.5 (1.5)	20–18–17	56.4 (1.4)
10–9–6	57.8 (1.5)	20–19–16	55.8 (1.4)
10–9–8	56.2 (1.5)	20–19–18	57.7 (1.4)

At Equatorial Atoms, One Leg across Equatorial Belt

3–2–6	59.2 (1.6)	13–12–16	58.3 (1.6)
5–2–9	59.4 (1.5)	15–12–19	58.9 (1.5)
4–3–7	59.0 (1.5)	14–13–17	60.4 (1.6)
2–3–6	58.7 (1.6)	12–13–16	60.7 (1.5)
5–4–8	58.4 (1.6)	15–14–18	58.8 (1.5)
3–4–7	58.5 (1.5)	13–14–17	59.9 (1.6)
2–5–9	58.4 (1.5)	12–15–19	60.3 (1.5)
4–5–8	59.1 (1.6)	14–15–18	59.8 (1.5)
7–6–3	57.1 (1.5)	17–16–13	59.9 (1.6)
9–6–2	59.2 (1.5)	19–16–12	58.4 (1.4)
8–7–4	59.4 (1.6)	18–17–14	59.6 (1.5)
6–7–3	58.5 (1.5)	16–17–13	59.6 (1.6)
9–8–5	59.3 (1.6)	19–18–15	59.3 (1.4)
7–8–4	60.1 (1.5)	17–18–14	58.0 (1.4)
6–9–2	59.5 (1.5)	16–19–12	59.2 (1.5)
8–9–5	56.9 (1.5)	18–19–15	60.4 (1.5)

At Equatorial Atoms, Both Legs across Equatorial Belt

6–2–9	61.3 (1.6)	16–12–19	62.4 (1.5)
6–3–7	64.4 (1.6)	16–13–17	60.6 (1.5)
7–4–8	60.5 (1.5)	17–14–18	62.5 (1.5)
8–5–9	63.8 (1.7)	18–15–19	60.3 (1.5)
2–6–3	62.2 (1.6)	12–16–13	61.0 (1.6)
3–7–4	62.5 (1.6)	13–17–14	59.6 (1.6)
4–8–5	62.5 (1.6)	14–18–15	61.4 (1.5)
2–9–5	62.2 (1.6)	12–19–15	60.8 (1.6)

Sum of Angles at Each Vertex

1	264.3 (3.3)	11	263.2 (3.6)
2	293.8 (3.4)	12	292.4 (3.5)
3	295.0 (3.5)	13	297.5 (3.5)
4	292.3 (3.4)	14	294.5 (3.5)
5	295.3 (3.5)	15	295.2 (3.5)
6	290.8 (3.4)	16	293.4 (3.4)
7	295.2 (3.5)	17	292.2 (3.4)
8	295.6 (3.4)	18	292.6 (3.2)
9	292.6 (3.4)	19	293.9 (3.3)
10	265.3 (3.3)	20	265.2 (3.1)

Pyramid Base Angles

5–2–3	88.7 (1.8)	15–12–13	88.8 (1.8)
2–3–4	90.5 (1.8)	12–13–14	91.9 (1.8)
3–4–5	89.9 (1.8)	13–14–15	89.4 (1.8)
4–5–2	90.9 (1.8)	14–15–12	89.9 (1.8)
9–6–7	88.7 (1.7)	19–16–17	90.2 (1.7)
6–7–8	90.7 (1.7)	16–17–18	89.9 (1.7)
7–8–9	90.4 (1.7)	17–18–19	90.3 (1.7)
8–9–6	90.3 (1.7)	18–19–16	89.6 (1.6)

Terminal Hydrogen Angles

61–1–2	123 (7)	71–11–12	126 (7)
61–1–3	133 (7)	71–11–13	130 (7)
61–1–4	135 (7)	71–11–14	134 (7)
61–1–5	126 (7)	71–11–15	130 (7)
62–2–1	127 (7)	72–12–11	118 (7)
62–2–3	141 (7)	72–12–13	137 (7)
62–2–5	127 (7)	72–12–15	125 (7)
62–2–6	116 (7)	72–12–16	126 (7)
62–2–9	109 (7)	72–12–19	118 (7)
63–3–1	117 (8)	73–13–11	116 (8)
63–3–2	131 (8)	73–13–12	131 (7)
63–3–4	129 (8)	73–13–14	128 (7)
63–3–6	122 (8)	73–13–16	121 (7)
63–3–7	121 (8)	73–13–17	119 (8)
64–4–1	119 (8)	74–14–11	124 (7)
64–4–3	137 (7)	74–14–13	135 (7)
64–4–5	126 (8)	74–14–15	131 (7)
64–4–7	124 (8)	74–14–17	116 (7)
64–4–8	117 (8)	74–14–18	115 (7)
65–5–1	119 (7)	75–15–11	119 (7)
65–5–4	127 (7)	75–15–14	135 (7)
65–5–2	134 (7)	75–15–12	128 (7)
65–5–8	118 (7)	75–15–18	121 (7)
65–5–9	122 (7)	75–15–19	117 (7)
65–5–10	125 (7)	76–16–20	115 (7)
66–6–9	139 (7)	76–16–19	133 (7)
66–6–7	128 (7)	76–16–17	126 (7)
66–6–2	120 (7)	76–16–12	125 (7)
66–6–3	113 (7)	76–16–13	123 (7)
67–7–10	127 (7)	77–17–20	116 (8)
67–7–6	129 (7)	77–17–16	137 (8)
67–7–8	138 (7)	77–17–18	123 (7)
67–7–3	110 (7)	77–17–13	128 (8)
67–7–4	115 (7)	77–17–14	117 (8)
68–8–10	118 (7)	78–18–20	123 (7)
68–8–7	132 (7)	78–18–17	130 (7)
68–8–9	129 (7)	78–18–19	134 (7)
68–8–4	121 (7)	78–18–14	116 (7)
68–8–5	119 (7)	78–18–15	119 (7)
69–9–10	124 (8)	79–19–20	113 (7)
69–9–8	135 (7)	79–19–18	131 (7)
69–9–6	131 (7)	79–19–16	127 (7)
69–9–2	116 (7)	79–19–12	122 (7)
69–9–5	118 (8)	79–19–15	126 (7)

Triethylammonium Angles

24–23–25	108 (2)	31–30–32	119 (2)
24–23–26	110 (2)	31–30–33	109 (3)
25–23–26	120 (3)	32–30–33	108 (3)
23–24–27	116 (3)	30–31–34	116 (3)
23–25–28	116 (3)	30–32–35	113 (3)
23–26–29	114 (3)	30–33–36	122 (4)
38–37–39	115 (3)	41–37–42	108 (5)
38–37–40	110 (3)	41–37–43	105 (4)
39–37–40	106 (3)	42–37–43	121 (5)
37–38–44	109 (4)	37–41–44	113 (5)
37–39–45	112 (4)	37–42–45	125 (9)
37–40–46	111 (4)	37–43–46	107 (4)

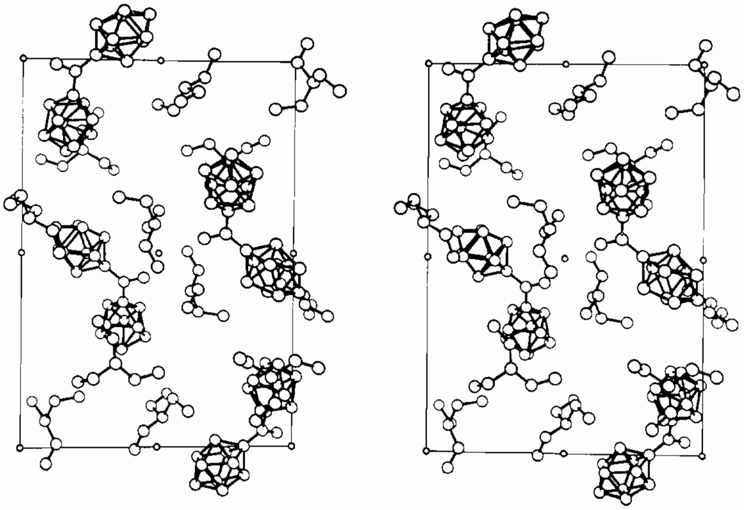

Figure 6.—Packing of the ions in $[(C_2H_5)_3NH]_3B_{20}H_{18}NO$. The origin is at the upper left, the *a* axis is vertical in the plane of the paper, the *b* axis is horizontal, and the *c* axis points upward out of the plane of the paper. Only the major component of the triethylammonium disorder is shown.

TABLE VIII

SHORTEST INTERIONIC NONBONDED CONTACTS (Å)[a]

22s–97	1.9 (2)	66–94	2.2 (3)	75g–137	2.2 (3)
75s–113	1.9 (3)	69–87	2.2 (3)	15s–113	2.3 (2)
61–119	2.1 (3)	88–129	2.2 (3)	2–81	2.5 (2)
75g–116	2.1 (3)	79s–113	2.2 (3)	3–81	2.5 (2)
63–81	2.2 (3)				

[a] Atoms transformed by symmetry operations are labeled with s for screw axis or g for glide plane; estimated errors are in parentheses. All of these contacts involve at least one hydrogen atom.

and disorder but average to 1.54 (8) Å for N–C and 1.46 (12) Å for C–C. The short nonbonded contact between H(75s) and H(113) probably results from errors and the fact that H(113) is a composite of two unresolved disordered positions. The packing of the ions with the minor disorder component omitted is shown in Figure 6. In this view the *a* axis is vertical, the *b* axis is horizontal, and the *c* axis is out of the plane of the paper.

The visible light absorption spectrum of $B_{20}H_{18}NO^{3-}$, as the K^+ salt to avoid triethylammonium hydrogen-bonding effects, displays the same two maxima in all eight solvents tested (Table IX). An intense peak at long wavelength (λ_{max} 5820 Å, ϵ 26,500 l. mol^{-1} cm^{-1} in pure acetonitrile[4]) generally shifts toward the red and its width at 90% of peak absorbance increases as solvent polarity, measured by the Kosower *Z* value,[19] decreases. However, in the π-bonded solvents acetonitrile, dimethyl sulfoxide, and acetone, the peak is sharpened, especially on the long-wavelength side. Such sharpening may be a result of π–π interaction and is another factor to be considered in comparing spectra of substituted $B_{20}H_{18}NO^{3-}$ ions[4] taken in acetonitrile. The peak at shorter wavelength is less intense (λ_{max} 4130 Å, ϵ approximately 4500 l. mol^{-1} cm^{-1} in 1% aqueous acetonitrile) but displays a similar red shift with decreasing *Z*. While peak width shows no change with *Z*, it is symmetrically narrowed in π-bonded solvents.

TABLE IX

SOLVENT DEPENDENCE OF ABSORPTION MAXIMA[a] AND WIDTHS

Solvent: 1% water plus	Kosower[b] *Z*	5800-Å peak pos	Width at 90% abs	4100-Å peak pos	Width at 90% abs
Water	94.6	5743	−238, +225	4070	−166, +178
Methanol	83.8	5721	−238, +232	4091	−157, +166
Ethanol	79.9	5770	−250, +228	4118	−158, +172
tert-Butyl alcohol	72.5	5967	−357, +284	4291	−183, +157
Dioxane	(68)	5942	−457, +413	4200	−175, +143
CH_3CN	(71)	5760	−212, +146	4130	−140, +135
$(CH_3)_2SO$	(72)	5803	−212, +135	4151	−145, +135
$(CH_3)_2CO$	68.1	5711	−184, +131	4134	−148, +141

[a] Reproducibility of peak position is 5–10 Å except 20 Å for *tert*-butyl alcohol and dioxane. Measurements were taken at 26 (2)°. [b] Interpolated or estimated from ref 19. Estimates are given in parentheses.

Attempted preparations of equatorially bonded isomers of $B_{20}H_{18}NO^{3-}$ by irradiation at 5800 Å (9 × 10^2 einsteins delivered to 2 × 10^{-3} formula weight), at 4000 Å (2 × 10^2 einsteins, 2 × 10^{-3} formula weight), and at 3150 Å (3 einsteins, 10^{-4} formula weight) produced no change in the uv–visible absorption spectrum. Also, no spectral evidence of complex formation with transition metals could be obtained. Aqueous solutions 5 × 10^{-5} *M* in $K_3B_{20}H_{18}NO$ and about 0.1 *M* in $MnSO_4$, $CoSO_4$, $FeSO_4$, $Fe(NO_3)_3$, or $Fe_2(SO_4)_3$ + H_2SO_4 compared with solutions of the same components in separate cuvettes showed only insignificant changes attributable to concentration differences.

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