refined with anisotropic displacement parameters; the H atoms were located in calculated positions with isotropic displacement parameters equal to the isotropic equivalent of their carrier C atom (C—H = 0·96 Å). The refinement converged at $R = 0·052$, $wR = 0·071$ ($g = 0·0012$) and $S = 1·49$ with 109 parameters for the α-form and at $R = 0·047$, $wR = 0·051$ ($g = 0·0005$) and $S = 1·26$ with 109 parameters for the β-form, with $w^{-1} = [\sigma^2(F) + g(F)^2]$ and $(\Delta/\sigma)_{\max} = 0·005$ and 0·001 for the α- and β-forms, respectively, and $(\Delta\rho)_{\max}$, $(\Delta\rho)_{\min} = +0·26$, $-0·26$ and $+0·21$, $-0·22$ e Å^{-3} for the α- and β-forms, respectively. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations, including the drawing of diagrams, were performed on a Nova 4X computer using *SHELXTL* (Sheldrick, 1983).

Table 1 lists the atom coordinates and Table 2 the bond lengths and angles.* Fig. 1 shows the structure and atom labelling. The two forms differ only in the crystal packing which shows the common herringbone pattern in both cases but with different interlayer stacking. Fig. 2 highlights the stacking differences and shows the overlap of adjacent molecules projected through their mean planes which are separated by 3·428 and 3·458 Å for the α- and β-forms respectively.

Related literature. The synthesis and a diruthenium(II) complex of the title compound have been described by Downard, Honey, Phillips & Steel (1991) and the structure and conformation of 2,2′-bipyridine by Almenningen, Bastiansen, Gundersen & Samdal (1989). For two preparations and the crystal structure of 2,2′:6′,2″:6″,2‴-quaterpyridine see Constable, Elder, Healy & Tocher (1990) and Uenishi, Tanaka, Wakabayashi, Oae & Tsukube (1990), and for 4,4′:2′,2″:4″,4‴-quaterpyridine see Morgan & Baker (1990). A survey of related binucleating ligands is given by Steel (1990).

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* Lists of structure factors, anisotropic displacement parameters and calculated H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54200 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1991). C**47**, 2249–2252

Structures of Two *N*-Acyl Triethyl Ammonium Salts and One Simple Triethyl Ammonium Salt

BY JOSEPH A. KING JR AND GAROLD L. BRYANT JR

General Electric Company, Corporate Research and Development, Schenectady, NY 12301, *USA*

(*Received* 20 *November* 1990; *accepted* 29 *April* 1991)

Abstract. *N*-Benzoyl-*N*-triethylammonium tetraphenylborate (I), $C_{13}H_{20}NO^{+}.C_{24}H_{20}B^{-}$, $M_r = 525·5$, monoclinic, $P2_1$, $a = 9·666$ (2), $b = 9·933$ (2), $c = 16·023$ (3) Å, $\beta = 106·01$ (2)°, $V = 1478·7$ Å^3, $Z = 2$, $D_x = 1·180$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·64$ cm^{-1}, $F(000) = 564$, $T = 191$ K, $R = 0·0483$ for 2844 unique reflections with $I > 4\sigma(I)$. *N*-Triethyl-*N*-phenoxycarbonylammonium tetrafluoroborate (II), $C_{13}H_{20}NO_2{}^{+}.BF_4^{-}$, $M_r = 309·1$, monoclinic, $P2_1/c$, $a = 9·196$ (3), $b = 13·442$ (3), $c = 12·718$ (3) Å, $\beta = 104·46$ (2)°, $V = 1522·3$ Å^3, $Z = 4$, $D_x = 1·349$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1·14$ cm^{-1}, $F(000) = 648$, $T = 205$ K, $R = 0·0477$ for 2853 unique reflections with $I > 3\sigma(I)$. Triethylammonium tetraphenylborate (III), $C_6H_{15}N^{+}.C_{24}H_{20}B^{-}$, $M_r = 421·4$, monoclinic, $P2_1/m$, $a = 9·291$ (4), $b = 14·368$ (13), $c = 9·559$ (7) Å, $\beta = 109·76$ (5)°, $V = 1200·9$ Å^3, $Z = 2$, $D_x = 1·165$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·61$ cm^{-1}, $F(000) = 456$, $T = 208$ K, $R = 0·0743$ for 1274 unique reflections with $I > 4\sigma(I)$. (I) and (II) are *N*-acyl ammonium salts; (III) is an ammonium

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Table 1. *Experimental details*

	(I)	(II)	(III)
Crystal habit	Prism	Parallelepiped	Parallelepiped
Size (mm)	0·5 × 0·4 × 0·20	0·42 × 0·24 × 0·10	0·48 × 0·30 × 0·10
Lattice parameters			
Number of reflections	22	40	28
2θ range (°)	5·9–27·9	13·4–26·8	7·5–23·0
Reflection range			
h	−4 to 12	−2 to 12	−12 to 0
k	−12 to 12	−17 to 17	0 to 18
l	−20 to 20	−16 to 16	−11 to 12
Max. $\sin\theta/\lambda$ (Å^{-1})	0·650	0·650	0·650
Check reflections	$\bar{4}00$ $20\bar{4}$ 115	$\bar{1}20$ $\bar{2}2\bar{2}$ $\bar{2}05$	$\bar{3}01$ 002 $\bar{2}\bar{2}1$
variation (%)	5 3 5	5 6 10	6 7 11
Reflections			
Collected	3795	7999	3038
Unique observed	2844	2853	1274
R_{int}	0·012	0·026	0·036
Observed criterion	$I > 4\sigma(I)$	$I > 2\sigma(I)$	$I > 4\sigma(I)$
Number of parameters	362	191	157
R, *wR*	0·047, 0·050	0·048, 0·067	0·074, 0·075
S	1·37	1·43	1·59
Secondary-extinction parameter† (χ)	0·0017 (4)	0·0023 (5)	0·0032 (10)
Weighting factor‡ (*g*)	0·0004	0·0011	0·0006
Min. and max. Fourier difference peaks (e Å^{-3})	−0·19, 0·27	−0·27, 0·35	−0·34, 0·43
Max. Δ/σ	0·703	0·257	0·380

† $F^* = F[1 + 0{\cdot}002\chi F^2/\sin(2\theta)]^{-1/4}$.
‡ $w^{-1} = \sigma^2(F) + gF^2$.

salt for comparative purposes. The N—$C_{carbonyl}$ bond lengths are 1·548 (I) and 1·497 Å (II). The O—C bond lengths of the carbonyl moieties are 1·186 (I) and 1·180 Å (II). The C_{aryl}—$C_{C=O}$—N angle is 118·3 (3)° in (I), while the O—$C_{C=O}$—N angle is 108·8 (1)° in (II). The $O_{C=O}$—$C_{C=O}$—N angles are 117·1 (3) (I) and 122·4 (2)° (II), respectively. In all three of the structures, the ethyl groups of the ammonium moieties adopt identical conformations. In (I) and (II), one ethyl group in each compound lies essentially within the plane defined by the N atom and the carbonyl group with the methyl group anti-parallel to the carbonyl O atom. The remaining two ethyl groups are oriented in a plane – through the nitrogen – which is perpendicular to the nitrogen–carbonyl group plane; the methyl group on each of these perpendicular ethyl moieties is oriented towards the O atom of the carbonyl functionality. One of the ethyl groups in (III) is disordered; this group was refined isotropically assuming two slightly different conformations. The site occupancy for atoms C17 and C18 was refined to 0·52 (1), with atoms C19 and C20 being occupied for the remainder. It is of interest to note that these triethyl ammonium moieties adopt similar conformations independent of either the anions or the appended group.

Experimental. Compound (I) obtained by reaction of triethylamine, benzoyl chloride, and silver tetrafluoroborate, followed by sodium tetraphenylborate

Table 2. *Atom coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3) *for* (I)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
B(1)	−1783 (3)	0 (2)	−2685 (2)	16 (1)
O(1)	4540 (3)	847 (5)	1743 (1)	31 (1)
N(1)	6524 (3)	222 (4)	2885 (2)	17 (1)
C(1)	6090 (3)	−677 (5)	1307 (2)	21 (1)
C(2)	5117 (4)	−1694 (5)	921 (2)	30 (1)
C(3)	5409 (4)	−2508 (5)	297 (2)	36 (1)
C(4)	6648 (4)	−2335 (5)	45 (2)	34 (1)
C(5)	7599 (4)	−1329 (6)	413 (2)	36 (1)
C(6)	7339 (4)	−495 (5)	1043 (2)	31 (1)
C(7)	5623 (3)	215 (5)	1922 (2)	20 (1)
C(8)	6488 (3)	−1218 (5)	3219 (2)	24 (1)
C(9)	5034 (4)	−1653 (5)	3290 (3)	34 (1)
C(10)	5829 (3)	1220 (5)	3366 (2)	23 (1)
C(11)	6605 (4)	1337 (5)	4323 (2)	32 (1)
C(12)	8087 (3)	564 (5)	2975 (2)	22 (1)
C(13)	8278 (4)	1946 (5)	2628 (2)	28 (1)
C(14)	1526 (3)	1642 (5)	−2662 (2)	17 (1)
C(15)	−1317 (3)	2353 (5)	−1880 (2)	23 (1)
C(16)	−1262 (4)	3754 (5)	−1843 (2)	27 (1)
C(17)	−1387 (3)	4509 (5)	−2586 (2)	27 (1)
C(18)	−1580 (4)	3840 (5)	−3368 (2)	24 (1)
C(19)	−1663 (3)	2437 (5)	−3399 (2)	21 (1)
C(20)	−3488 (3)	−153 (5)	−2712 (2)	19 (1)
C(21)	−3935 (4)	−86 (5)	−1958 (2)	27 (1)
C(22)	−5376 (4)	−46 (5)	−1961 (3)	35 (1)
C(23)	−6438 (4)	−57 (5)	−2743 (3)	37 (1)
C(24)	−6041 (4)	−138 (5)	−3506 (3)	34 (1)
C(25)	−4592 (3)	−179 (5)	−3489 (2)	25 (1)
C(26)	−1432 (3)	−746 (5)	−3521 (2)	18 (1)
C(27)	−2095 (3)	−1968 (5)	−3856 (2)	23 (1)
C(28)	−1746 (4)	−2663 (5)	−4523 (2)	28 (1)
C(29)	−702 (4)	−2174 (5)	−4881 (2)	29 (1)
C(30)	−9 (3)	−987 (5)	−4567 (2)	26 (1)
C(31)	−364 (3)	−297 (5)	−3897 (2)	20 (1)
C(32)	−713 (3)	−751 (5)	−1836 (2)	18 (1)
C(33)	−1079 (3)	−1974 (5)	−1519 (2)	19 (1)
C(34)	−138 (3)	−2673 (5)	−843 (2)	23 (1)
C(35)	1221 (3)	−2161 (5)	−453 (2)	24 (1)
C(36)	1623 (3)	−950 (5)	−743 (2)	25 (1)
C(37)	672 (3)	−259 (5)	−1423 (2)	21 (1)

Table 3. *Atom coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3) *for* (II)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
B(1)	2543 (2)	5661 (2)	1582 (2)	31 (1)
F(1)	1671 (1)	6189 (1)	709 (1)	41 (4)
F(2)	3706 (1)	5194 (1)	1255 (1)	46 (4)
F(4)	3161 (1)	6319 (1)	2419 (1)	45 (1)
F(3)	1687 (2)	4958 (1)	1937 (1)	62 (1)
N(1)	2625 (2)	2781 (1)	−1034 (1)	25 (1)
O(1)	3039 (1)	1139 (1)	−570 (1)	30 (1)
O(2)	1751 (1)	2057 (1)	377 (1)	31 (1)
C(1)	2934 (2)	271 (1)	64 (1)	28 (1)
C(2)	3904 (2)	184 (1)	1071 (1)	31 (1)
C(3)	3811 (2)	−676 (1)	1663 (2)	35 (1)
C(4)	2771 (2)	−1407 (1)	1233 (2)	35 (1)
C(5)	1837 (2)	−1295 (1)	204 (2)	39 (1)
C(6)	1904 (2)	−440 (1)	−399 (2)	36 (1)
C(7)	2400 (2)	1949 (1)	−307 (1)	25 (1)
C(8)	1979 (2)	2470 (1)	−2220 (1)	31 (1)
C(9)	330 (2)	2231 (2)	−2485 (1)	37 (1)
C(10)	1851 (2)	3710 (1)	−741 (1)	28 (1)
C(11)	1990 (2)	4615 (1)	−1414 (2)	39 (1)
C(12)	4313 (2)	2944 (1)	−869 (2)	31 (1)
C(13)	5143 (2)	3131 (2)	299 (2)	37 (1)

Table 4. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$) *for* (III)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N(1)	1178 (5)	2500	3329 (5)	33 (2)
B(1)	−1801 (7)	2500	6921 (7)	28 (2)
C(1)	−2537 (4)	1585 (3)	7460 (4)	29 (1)
C(2)	−1879 (5)	1192 (3)	8855 (4)	44 (2)
C(3)	−2528 (6)	437 (4)	9348 (5)	56 (2)
C(4)	−3895 (6)	59 (3)	8451 (6)	48 (2)
C(5)	−4591 (5)	443 (3)	7065 (6)	48 (2)
C(6)	−3933 (5)	1189 (3)	6599 (5)	41 (2)
C(7)	85 (6)	2500	7590 (5)	23 (2)
C(8)	942 (4)	1692 (3)	7819 (5)	44 (2)
C(9)	2536 (5)	1680 (3)	8229 (5)	46 (2)
C(10)	3337 (7)	2500	8440 (6)	34 (2)
C(11)	−2182 (6)	2500	5096 (6)	39 (3)
C(12)	−2322 (5)	1679 (4)	4280 (5)	55 (2)
C(13)	−2590 (5)	1680 (5)	2755 (5)	76 (3)
C(14)	−2733 (8)	2500	1988 (8)	85 (5)
C(15)	3509 (6)	2500	2517 (6)	39 (2)
C(16)	1783 (8)	2500	2054 (7)	84 (4)
C(17)	1464 (9)	1591 (6)	3998 (10)	28 (2)
C(18)	890 (10)	842 (6)	2795 (10)	44 (3)
C(19)	1778 (11)	1764 (7)	4629 (12)	36 (3)
C(20)	1447 (11)	736 (7)	4109 (12)	51 (3)

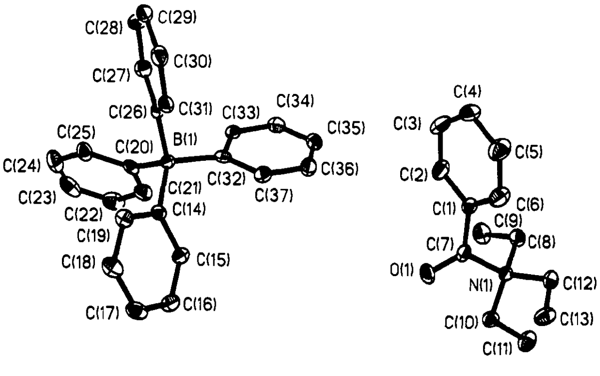

Fig. 1. Thermal-ellipsoid (50% probability) plot of (I).

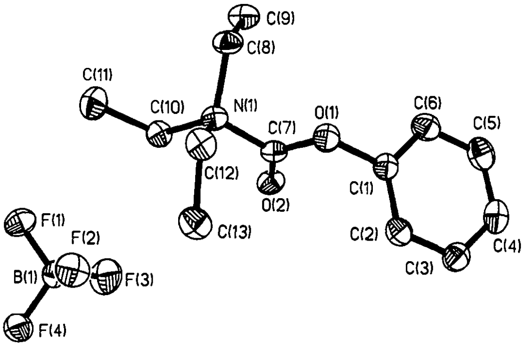

Fig. 2. Thermal-ellipsoid (50% probability) plot of (II).

in an acetonitrile solution. Compound (I) solution recrystallized from a CH_2Cl_2/ether solution at 253 K. Compound (II) obtained by reaction of triethylamine, phenylchloroformate and silver tetrafluoroborate in an acetonitrile solution. Compound (II) recrystallized from CH_2Cl_2/ether solution at 253 K. Compound (III) obtained by reaction of H_2O on (I) in acetonitrile. Compound (III) recrystallized from CH_2Cl_2/ether at 253 K. Compounds (I), (II) and (III) sealed in glass capillary under dry nitrogen due to their moisture sensitivity. Siemens *R*3*m*/*V* upgrade of Nicolet *P*3*F* automated diffractometer, 2θ–θ scan with variable scan speeds. D_m values not determined. Both structures were solved by direct methods using

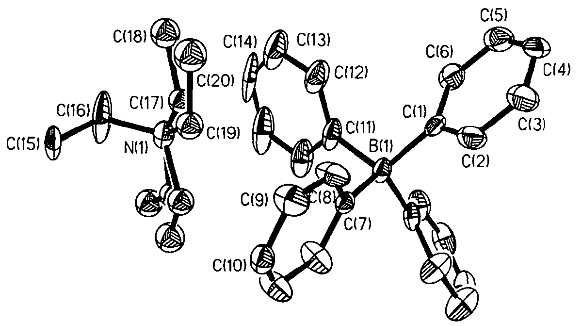

Fig. 3. Thermal-ellipsoid (50% probability) plot of (III), showing disorder for one of the ethyl groups.

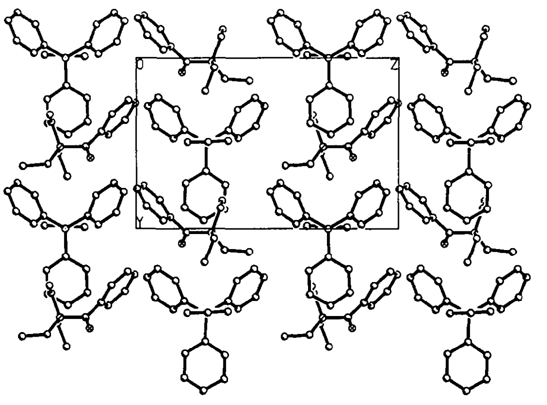

Fig. 4. Projected packing plot of (I) viewed down the *a* axis.

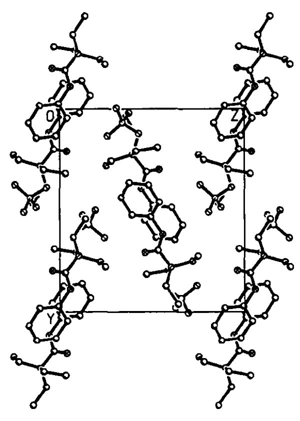

Fig. 5. Projected packing plot of (II) viewed down the *a* axis.

the *SHELXTL-Plus* program package (Sheldrick, 1988) on a MicroVAX II computer. H atoms were placed in idealized positions and constrained to have C—H = 0·96 Å and isotropic thermal parameters, U = 0·08 Å^2. All non-H atoms treated as anisotropic, except the disordered atoms in (III). No absorption correction necessary for any of the structures. Details of the data collection are given in Table 1. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates for (I), (II) and (III) are contained in Tables 2, 3 and 4.* Figs. 1, 2 and 3 illustrate the molecules with the numbering scheme employed. Figs. 4, 5 and 6 illustrate the projected packing of the molecules viewed down the *a* axis.

Related literature. For additional information on related *N*-acyl ammonium salt reactions and chemistry see Gresser & Jencks (1977), Jencks & Gilchrist (1968), Kim, Lee & Kim (1985), Bayliss, Homer & Shepherd (1990) and Brunelle, Boden & Shannon (1990).

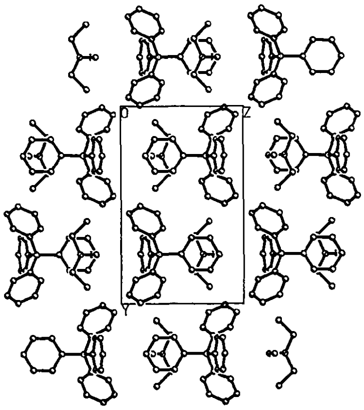

Fig. 6. Projected packing plot of (III) viewed down the *a* axis.

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54207 (54 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1991). C**47**, 2252–2254

Structure of 1,3-Dinitronaphthalene

By Herman L. Ammon

Department of Chemistry and Biochemistry, University of Maryland, College Park, MD 20742, *USA*

(*Received* 14 *January* 1991; *accepted* 15 *March* 1991)

Abstract. $C_{10}H_6N_2O_4$, M_r = 218·2, triclinic, $P\bar{1}$, a = 7·5552 (8), b = 7·763 (1), c = 9·073 (1) Å, α = 110·742 (9), β = 99·570 (9), γ = 100·309 (9)°, V = 474·1 (2) Å^3, D_x = 1·528 g cm^{-3}, Z = 2, Cu $K\alpha$ (λ = 1·54178 Å, graphite monochromator) μ = 10·5 cm^{-1}, F(000) = 224, T = 293 K, final R = 0·064, wR = 0·092 for 1213 reflections with $I > 3\sigma(I)$. The naphthalene ring is planar with an average deviation of 0·004 Å from the ten-atom least-squares plane. The C—C distances show the typical naphthalene alternation: C1—C2, C3—C4, C5—C6 and C7—C8 average 1·359 Å; C2—C3 and C6—C7 average 1·391 Å. The C1 nitro group is twisted out of the ring plane by approximately 37° to relieve non-bonded interactions between an oxygen and the C8—H8 region.

Experimental. Commercial sample (Aldrich Chemical Co.), orange-brown needle-like crystals from cyclohexane/1,2-dichloroethane by vapor diffusion, 0·2 × 0·23 × 0·46 mm specimen; Enraf–Nonius CAD-4 diffractometer; cell parameters and crystal orientation from 25 automatically centered reflections in the range 7·1 < θ < 43·2°; 2θ–θ scans over $\Delta\theta$ range of 1·5 (1·1 + 0·14tanθ)°; variable θ scan speed of 1·18–5·49° min^{-1}; each scan recorded in 96 steps with two outermost 16-step intensity blocks used for background determination; diffractometer controlled with Digital Equipment Corporation MicroVaxII computer and standard Enraf–Nonius programs (version 5·0); nine standard reflections monitored at 1 h intervals of X-ray exposure, −2·4 to 0·4% intensity variation, −0·9% average, correc-

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