Perhaps the most useful correlation can be made with the negative charge of the sulfur atom. This might account also for the spectrochemical difference between bis(diethyldithiophosphato)nickel(II)[8] and bis(diphenyldithiophosphinato)nickel(II) since, in the former compound, one has the inductive effect of the oxygen which draws electrons from the sulfur atom. The only compound whose data do not fit the curve of Figure 3 well is bis(diethyldithioxanthate)nickel(II);[4] it should also be noted that the same compound behaves anomalously in that it has an unusually low spectrochemical position.

The C(1) and C(7) carbon atoms of the two phenyl groups bonded to the phosphorus atom (whose distances from the sulfur atoms are in agreement with the values reported in the literature for other similar compounds[8]) are in such position as to form with the sulfur atoms the slightly distorted tetrahedral configuration around phosphorus.

As reported in Table V other least-squares planes were calculated and these pass precisely through the carbon atoms of the phenyl groups, which are approximately planar. The maximum deviations from the planes are 0.026 and 0.07 Å for planes II and III, respectively. The standard deviations for the carbon parameters (Table II) are naturally higher than those of the heavier atoms, but, even so, the bond angles and distances within the phenyl groups are reasonable with an average C–C bond length of 1.38 ± 0.04 Å and bond angles equal to 120 ± 3°.

The dihedral angles (Table V) between planes I, II, and III are: 53° 49′ (between I and II), 89° 49′ (between I and III), and 74° 6′ (between II and III).

It is worth noting that very recently a paper[24] reported preliminary data of the crystal structure determination of a compound very similar to ours, the only difference being that the phenyl groups are methyl substituted. The configuration around the nickel atom in this compound has been found to be planar also and the average bond lengths and angles are in good agreement with those reported here.

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The Crystal and Molecular Structure of Tetraphenylarsonium Triiodo(triphenylphosphine)nickelate(II), $[(C_6H_5)_4As][Ni(C_6H_5)_3PI_3]$[1a]

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The structure of tetraphenylarsonium triiodo(triphenylphosphine)nickelate(II), $[(C_6H_5)_4As][Ni(C_6H_5)_3PI_3]$, has been determined by single-crystal X-ray diffraction. The crystals are monoclinic, space group I2/c, with $a = 33.971 \pm 0.004$, $b = 14.992 \pm 0.002$, $c = 16.253 \pm 0.004$ Å, and $\beta = 92.06 \pm 0.05°$. For $Z = 8$ the measured and calculated densities are 1.73 ± 0.02 and 1.745 g/cm³, respectively. Counter data were collected for 2548 independent reflections by the stationary-crystal, stationary-counter technique. The structure was refined by least-squares methods to a conventional R value of 0.082. The pseudo-tetrahedral anion has approximately C_{3v} symmetry, excluding the phenyl groups, with an average Ni–I distance of 2.55 Å and an Ni–P distance of 2.28 Å. The tetraphenylarsonium ion is unremarkable (almost exactly tetrahedral angles at the arsenic atom) and possesses no over-all symmetry. Four nearest neighbor cations approach a given anion to within 6.6–8.0 Å (Ni–As distance). The proximity and mode of counterion approach is compared with earlier solution-state estimates based on pmr data.

Introduction

Not long ago in one of these laboratories it was discovered[2,3] that large proton chemical shifts occur for diamagnetic cations in the presence of certain paramagnetic anions as a result of a dipolar nucleus–unpaired electron interaction between ions in an ion pair. The phenomenon was first observed[2] in chloroform solutions of the tetra-*n*-butylammonium salts of the pseudo-tetrahedral anions $M(C_6H_5)_3PI_3^-$, M = Co, Ni. Such dipolar shifts (resonance frequency displacements from their diamagnetic positions) will generally occur when one of the ions in the ion pair exhibits significant magnetic anisotropy and there is a preferred relative orientation of the partners. Since the original discovery, such shifts have been observed[4–6] in a number of other systems where they are also related to ion association. The magnitude of the dipolar shift of a

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TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR $[(C_6H_5)_4As][Ni(C_6H_5)_3PI_3]$[a]

```
H,K=-20, 0
 L FOB FCA
10 91 -86

H,K=-18, 0
 L FOB FCA
10 48 -44

H,K=-16, 0
 L FOB FCA
 8 95 -92
10 69 -66

H,K=-14, 0
 L FOB FCA
 8 75 -67
10 0 -24*
12 35 32

H,K=-12, 0
 L FOB FCA
 6 180-170
 8 47 -49
10 99 97
12 66 69

H,K=-10, 0
 L FOB FCA
 6 0 4*
 8 151 151
10 81 75
12 19 -9

H,K= -8, 0
 L FOB FCA
 4 45 -44
 6 216 207
 8 215 211
10 58 50
12 0 12*

H,K= -6, 0
 L FOB FCA
 4 209 195
 6 55 47
 8 15 -9
10 24 -14
12 33 -31

H,K= -4, 0
 L FOB FCA
 2 0 13*
 4 373 358
 6 138 120*
 8 43 -64
10 141-145
12 84 -84

H,K= -2, 0
 L FOB FCA
 2 74 68
 4 165-132*
 6 98 -94
 8 47 -38
10 65 -62
12 33 -17

H,K= 0, 0
 L FOB FCA
 4 368-331
 6 265-260
 2 413 369
 8 70 -68
10 135 132
12 97 97

H,K= 0, 1
 L FOB FCA
 1 91 80
 3 104 92
 5 12 -33
 7 184-183
 9 92 -87
11 82 77
13 74 81

H,K= 0, 2
 L FOB FCA
 0 491-443
 2 311-286
 4 96 -78
 6 67 59
 8 37 -29
10 80 -73
12 54 -52

H,K= 0, 3
 L FOB FCA
 1 207 193
 5 210 200
 7 0 1*
 9 134-129
11 68 -82
13 40 -43

H,K= 0, 4
 L FOB FCA
 0 146-130
 2 32 -24
 4 238 207
 6 148 151
 8 89 87
10 59 60
12 34 24

H,K= 0, 5
 L FOB FCA
 1 59 -57
 3 129-117
 5 49 -39
 7 63 59
 9 35 35
11 27 29

H,K= 0, 6
 L FOB FCA
 0 224-193
 2 146-128
 6 79 76
 4 15 19
 8 54 -57
10 72 -79
12 54 -69

H,K= 0, 7
 L FOB FCA
 1 74 -80
 3 291-274
 7 34 27
 5 132-132
 9 93 83
11 49 58

H,K= 0, 8
 L FOB FCA
 0 218 207
 2 107 101
 4 118-107
 6 82 -75
 8 59 53
10 102 100

H,K= 0, 9
 L FOB FCA
 1 47 41
 3 70 65
 5 56 51
 7 27 18
 9 40 -40

H,K= 0, 10
 L FOB FCA
 0 139 129
 2 69 64
 4 45 -36
 6 94 -85
 8 94 -90

H,K= 0, 11
 L FOB FCA
 1 65 71
 3 95 92
 5 20 25

H,K= 0, 12
 L FOB FCA
 0 123-117
 2 49 -38
 4 61 51

H,K= 1, 1
 L FOB FCA
-12 86 76
-10 29 24
 -8 109-105
 -6 160-152
 -4 26 6
 -2 84 76
  0 0 1*
  2 14 10
  4 128-104
  6 56 61
  8 101 101
 10 72 -79
 12 62 -82

H,K= 1, 2
 L FOB FCA
-13 56 60
-11 114 110
 -9 105 109
 -7 0 -10*
 -5 274-246
 -3 441-390
 -1 37 30
  1 119 107
  3 123 117
  5 37 38
  7 82 81
  9 79 79
 11 31 -28
 13 63 -71

H,K= 1, 3
 L FOB FCA
-12 88 -89
-10 69 -66
 -8 79 65
 -6 67 65
 -4 135 117
 -2 0 -6*
  0 92 103
  2 246 213
  4 88 -79
  6 55 -57
  8 47 -48
 10 17 6
 12 77 77

H,K= 1, 4
 L FOB FCA
-13 20 -7
-11 19 4
 -9 61 -54
 -7 15 18
 -5 99 93
 -3 138 132
 -1 47 35
  1 223-194
  3 188-177
  5 81 -78
  7 71 66
  9 38 46
 11 26 11
 13 0 17*

H,K= 1, 5
 L FOB FCA
-12 60 -58
-10 107 106
 -8 168 165
 -6 42 46
 -4 149-167
 -2 182-175
  0 119-133
  2 77 -77
  4 120-117
  6 47 -37
  8 72 86
 10 109 108
 12 34 42

H,K= 1, 6
 L FOB FCA
-11 32 -45
 -9 0 2*
 -7 16 7
 -5 15 4
 -3 37 -35
 -1 47 48
  1 153 134
  3 226 213
  5 0 1*
  7 96-104
  9 76 -78
 11 20 15

H,K= 1, 7
 L FOB FCA
-10 27 -22
 -8 36 -22
 -6 0 -5*
 -4 64 64
 -2 102 95
  0 58 60
  2 64 64
  4 73 71
  6 33 32
  8 44 -51
 10 64 -73

H,K= 1, 8
 L FOB FCA
 -9 107-104
 -7 18 -8
 -5 77 69
 -3 123 122
 -1 22 -30
  1 152-142
  3 77 -72
  5 0 -7*
  7 25 3
  9 27 -22

H,K= 1, 9
 L FOB FCA
 -8 109 -96
 -6 102-101
 -4 30 -35
 -2 0 20*
  0 24 28
  4 17 -22
  6 0 1*
  8 34 -25

H,K= 1, 10
 L FOB FCA
 -7 71 65
 -5 77 -64
 -3 81 -74
 -1 40 38
  1 55 73
  3 65 58
  5 0 -12*
  7 20 5

H,K= 1, 11
 L FOB FCA
 -6 65 63
 -4 61 45
 -2 19 -25
  0 0 -18*
  4 51 46
  6 45 52

H,K= 1, 12
 L FOB FCA
 -3 0 -10*
 -1 20 -22
  1 79 -68
  3 57 -50

H,K= 2, 0
 L FOB FCA
  0 0 8*
  2 0 3*
  4 129 112
  6 56 -55
  8 65 -63
 10 63 -73
 12 33 -37

H,K= 2, 1
 L FOB FCA
-13 97-100
-11 95 -97
 -9 0 9*
 -7 212 201
 -5 219 198
 -3 153-133
 -1 350-321
  1 231-205
  3 308-271
  5 84 -76
  7 0 -1*
  9 36 44
 11 18 7
 13 0 -10*

H,K= 2, 2
 L FOB FCA
-12 33 -22
-10 89 85
 -8 212 206
 -6 63 58
 -4 167-152
 -2 263-250
  0 95-109
  2 118 113
  4 263 228
  6 42 54
  8 34 -26
 10 0 -12*
 12 19 -13

H,K= 2, 3
 L FOB FCA
-11 37 16
 -9 55 -61
 -7 44 46
 -5 250 227
 -3 147 134
 -1 144-133
  1 115-110
  3 130 107
  5 216 210
  7 238 236
  9 72 70
 11 41 -43
 13 35 -41

H,K= 2, 4
 L FOB FCA
-12 58 51
-10 85 -82
 -8 189-180
 -6 117-114
 -4 169 159
 -2 462 437
  0 172 184
  2 307-276
  4 383-355*
  6 59 -68
  8 183 187
 10 91 101
 12 28 -35

H,K= 2, 5
 L FOB FCA
-11 19 -7
 -9 17 14
 -7 61 -61
 -5 187-168
 -3 129-120
 -1 17 10
  1 98 91
  3 41 -42
  5 104-108
  7 167-167
  9 30 -31
 11 19 23

H,K= 2, 6
 L FOB FCA
-12 35 -27
-10 32 -15
 -8 17 5
 -6 69 62
 -4 56 46
 -2 74 71
  0 60 59
  2 0 3*
  4 73 -64
  6 121-122
  8 17 -30
 10 27 19
 12 41 38

H,K= 2, 7
 L FOB FCA
-11 78 82
 -9 59 48
 -7 0 -10*
 -5 48 -45
 -3 34 29
 -1 221 208
  1 183 163
  3 15 -16
  5 74 -73
  7 55 -56
  9 19 13
 11 49 51

H,K= 2, 8
 L FOB FCA
-10 51 42
 -8 70 54
 -6 0 0*
 -4 114-116
 -2 183-192
  0 84-100
  2 125 108
  4 182 162
  6 82 84
  8 37 -30
 10 40 -34

H,K= 2, 9
 L FOB FCA
 -9 56 -51
 -7 53 -51
 -5 31 -27
 -3 0 -6*
 -1 24 -31
  1 0 -15*
  3 57 54
  5 54 58
  7 33 -30
  9 66 -69

H,K= 2, 10
 L FOB FCA
 -8 70 21
 -6 0 -4*
 -4 37 -31
 -2 25 -22
  0 0 3*
  2 44 48
  4 52 50
  6 19 -18
  8 20 -33

H,K= 2, 11
 L FOB FCA
 -5 108 92
 -3 19 23
 -1 82 -77
  1 131-116
  5 56 55

H,K= 2, 12
 L FOB FCA
 -4 20 24
 -2 20 22
  0 0 -9*
  2 44 -38

H,K= 3, 1
 L FOB FCA
-12 50 57
-10 143 144
 -8 113 113
 -6 66 -62
 -4 307-275
 -2 12 -1
  0 43 46
  2 39 35
  4 46 -40
  6 106 93
  8 147 153
 10 48 56
 12 54 -57

H,K= 3, 2
 L FOB FCA
-13 109 -90*
-11 54 -53
 -9 90 80
 -7 80 82
 -5 52 44
 -3 62 50
 -1 49 44
  1 134 123
  3 99 94
  5 122-114
  7 131-131
  9 46 42
 11 103 103
 13 53 48

H,K= 3, 3
 L FOB FCA
-12 81 -85
-10 165-150
 -8 128-118
 -6 126 111
 -4 140 139
 -2 10 6
  0 127 133
  2 147-125
  4 115-100
  6 118-106
  8 154-169
 10 0 -12*
 12 80 82

H,K= 3, 4
 L FOB FCA
-13 50 -49
-11 37 -34
 -9 51 52
 -7 139 128
 -5 136 129
 -3 24 -39
 -1 227-216
  1 251-241
  3 180-155
  5 53 44
  7 0 6*
  9 17 16
 11 37 44

H,K= 3, 5
 L FOB FCA
-12 66 63
-10 32 -20
 -8 50 -58
 -6 0 -1*
 -2 130 114
  0 46 -49
  2 0 7*
  4 123 108
  6 114 107
  8 17 -10
 10 55 -62
 12 28 -33

H,K= 3, 6
 L FOB FCA
-11 107 106
 -9 40 -33
 -7 236-228
 -5 210-193
 -3 58 60
 -1 272 259
  1 245 228
  3 97 84
  5 15 22
  7 17 27
  9 26 -34
 11 71 -75

H,K= 3, 7
 L FOB FCA
-10 39 -39
 -8 59 56
 -2 51 -57
  0 21 31
  2 47 -54
  4 52 -51
  6 104 -93
  8 36 -47
 10 19 10

H,K= 3, 8
 L FOB FCA
 -9 54 -56
 -7 0 -2*
 -5 38 43
 -3 0 -32*
 -1 103-106
  1 127-117*
  3 28 -18
  5 42 39
  7 54 48
  9 33 -37

H,K= 3, 9
 L FOB FCA
 -8 58 59
 -6 66 -60
 -2 17 32
  0 29 37
  2 0 8*
  4 0 -2*
  6 96 79
  8 126 131

H,K= 3, 10
 L FOB FCA
 -7 52 52
 -5 32 26
 -3 18 18
 -1 36 36
  1 62 59
  3 0 -15*
  7 0 -5*

H,K= 3, 11
 L FOB FCA
 -6 0 -12*
 -4 19 -19
 -2 96 -92
  0 50 -58
  2 0 18*

H,K= 3, 12
 L FOB FCA

 -3 0 -13*
 -1 52 -62
  1 49 -44
  3 28 13

H,K= 4, 0
 L FOB FCA
  0 393-442
  2 471-431
  4 403-346
  6 215 184
  8 175 170
 10 45 -42
 12 91 -90

H,K= 4, 1
 L FOB FCA
-13 40 -18
-11 120 108
 -7 0 1*
 -5 95 -86
 -3 147 132
 -1 216 210
  1 42 39
  3 431-399
  5 312-280
  7 195 172
  9 221 238
 11 114 123
 13 35 -43

H,K= 4, 2
 L FOB FCA
-12 63 61
-10 132 131
 -8 96 97
 -6 23 18
 -4 108-103
 -2 42 -40
  0 193 208
  2 128 124
  4 78 89
  6 134 116
  8 67 69
 10 49 40
 12 0 -6*

H,K= 4, 3
 L FOB FCA
-13 20 7
-11 95 -90
 -9 128-128
 -7 57 -84*
 -5 68 61
 -3 237 209
 -1 49 62
  1 18 -10
  3 224-195
  5 182-164
  7 37 -27
  9 24 8
 11 0 -10*
 13 41 33

H,K= 4, 4
 L FOB FCA
-12 44 -35
 -6 141-138
 -8 121-111
-10 53 -58
 -4 163-148
 -2 0 -16*
  0 153 155
  2 44 92
  4 86 -81
  6 223-203
  8 185-172
 10 47 -46
 12 52 48

H,K= 4, 5
 L FOB FCA
-11 43 43
 -9 35 33
 -7 55 49*
 -5 50 -41
 -3 13 -17
 -1 33 24
  1 79 79
  3 90 83
  5 36 41
  7 36 -27
  9 30 -31
 11 0 -5*

H,K= 4, 6
 L FOB FCA
-12 29 16
-10 38 37
 -8 52 56
 -6 0 -18*
 -4 21 28
 -2 85 78
  0 149 152
  2 105 102
  4 0 19*
  6 48 -37
  8 25 -37
 10 27 16

H,K= 4, 7
 L FOB FCA
-11 20 -5
 -9 26 24
 -7 75 69
 -5 0 10*
 -3 151-168
 -1 270-271
  1 106 -99
  3 209 182
  5 244 219
  7 43 39
  9 80 -87
 11 45 -45

H,K= 4, 8
 L FOB FCA
-10 20 -26
 -8 32 -36
 -6 43 47
 -4 0 4*
 -2 104-104
  0 137-156
  2 129-124
  4 29 26
  6 147 134
 10 64 -72

H,K= 4, 9
 L FOB FCA
 -9 0 14*
 -7 46 -29
 -5 72 -64
 -3 34 37
 -1 127 119
  1 84 84
  3 0 -8*
  5 80 -68
  9 20 17

H,K= 4, 10
 L FOB FCA
 -8 29 27
 -6 74 74
 -4 101 95
 -2 78 67
  0 44 -53
  2 112-100
  4 61 -50*

H,K= 4, 11
 L FOB FCA
 -5 0 11*
 -3 88 86
 -1 65 69
  1 78 -63
  3 157-130

H,K= 4, 12
 L FOB FCA
 -4 110-100
 -2 40 -39
  0 52 64
  2 104 86

H,K= 5, 1
 L FOB FCA
-12 57 -52
-10 45 -32
 -8 57 58
 -6 87 86
 -4 23 -8
 -2 57 -52
  0 54 -57
  2 0 8*
  4 0 5*
  6 0 8*
  8 15 6
 10 0 -4*
 12 43 28

H,K= 5, 2
 L FOB FCA
-13 20 15
-11 94 -91
 -9 226-225
 -7 158-155
 -5 164 153
 -3 313 287
 -1 21 33
  1 38 -34
  3 46 -46
  5 78 -74
  7 138-135
  9 103 -96
 11 32 -52
 13 0 27*

H,K= 5, 3
 L FOB FCA
-12 27 10
-10 50 47
 -8 16 25
 -6 42 -41
 -4 0 -1*
 -2 137 129
  0 58 -55
  2 37 -37
  4 184-166
  6 129-111
  8 50 45
 10 15 17
 12 19 -3

H,K= 5, 4
 L FOB FCA
-11 27 11
 -9 59 55
 -7 134 125
 -5 89 86
 -3 138-120
 -1 152-147
  1 20 -16
  3 133 115
  5 163 144
  7 98 96
  9 46 46
 11 19 -17

H,K= 5, 5
 L FOB FCA
-12 109 108
-10 18 7
 -8 165-168
 -6 214-206
 -4 0 -6*
 -2 54 47
  0 153 162
  2 119 116
  4 158 152
  6 146 187
  8 45 43
 10 111 -97
 12 75 -89

H,K= 5, 6
 L FOB FCA
-11 28 16
 -9 18 25
 -7 58 -58
 -5 175-169
 -3 0 -9*
 -1 108 106
  1 0 1*
  3 29 -57
  5 136-130
  7 75 -70
  9 26 27
 11 0 -7*

H,K= 5, 7
 L FOB FCA
-10 0 -14*
 -8 67 61
 -6 47 50
 -4 39 -39
 -2 30 -44
  0 33 -30
  2 45 -46
  4 126-121
  6 130-114
  8 0 -5*

H,K= 5, 8
 L FOB FCA
 -9 108 106
 -7 89 89
 -5 0 3*
 -3 59 -61
 -1 0 -19*
  1 0 15*
  3 57 56
  5 107 96
  7 58 46

H,K= 5, 9
 L FOB FCA
 -8 48 35
 -6 132 121
 -4 125 124
 -2 24 -22
  0 81 -81
  2 0 -25*
  4 56 51
  6 56 56

H,K= 5, 10
 L FOB FCA
 -7 108-101
 -5 0 -2*
 -3 41 29
 -1 47 43
  1 60 -61
  3 105-103

H,K= 5, 11
 L FOB FCA
 -6 35 -17
 -4 103 -97
 -2 54 -59
  0 42 44
  2 41 40
  4 52 -45

H,K= 5, 12
 L FOB FCA
 -3 64 61
 -1 0 34*
  1 40 38
  3 50 44

H,K= 6, 0
 L FOB FCA
 -2 291 271

  0 36 25
  2 525-492
  4 439-400
  6 110-106
  8 98 92
 10 182 163

H,K= 6, 1
 L FOB FCA
-13 102 109
-11 127 123
 -9 80 81
 -7 0 -2*
 -5 67 -59
 -3 127-119
 -1 115 105
  1 154 143
  3 251 235
  5 209 193
  7 108 100
  9 40 52
 13 20 -11

H,K= 6, 2
 L FOB FCA
-12 33 34
-10 25 -3
 -8 58 -53
 -6 140-140
 -4 169-162
 -2 179 162
  0 197 215
  2 171 161
  4 70 61
  6 56 -48
  8 35 -40
 10 18 -8

H,K= 6, 3
 L FOB FCA
-13 99 -95
-11 76 -70
 -9 0 -7*
 -7 0 6*
 -5 211-189
 -3 231-224
 -1 0 12*
  1 319 308
  3 210 194
  5 198-183
  7 410-396
  9 203-190

H,K= 6, 4
 L FOB FCA
-12 28 3
-10 36 -25
 -8 59 54
 -6 176 173
 -4 27 13
 -2 302-279
  0 210-243
  2 25 19
  4 242 226
  6 146 137
  8 0 -13*
 10 106 -93

H,K= 6, 5
 L FOB FCA
-11 58 51
 -9 0 -13*
 -7 48 -49
 -5 107 102
 -3 213 197
 -1 146 136
  1 111-111
  3 120-117
  5 98 91
  7 231 219
  9 76 69

H,K= 6, 6
 L FOB FCA
-10 33 32
 -8 55 59
 -6 39 42
 -4 45 -46
 -2 59 -59
  0 68 -65
  2 0 -6*
  4 0 23*
  6 0 19*
  8 31 27

H,K= 6, 7
 L FOB FCA
-11 0 -5*
 -9 0 6*
 -7 25 26
 -5 65 62
 -3 38 -34
 -1 186-184*
  1 225-226
  3 158-151
  5 64 59
  7 106 98

H,K= 6, 8
 L FOB FCA
-10 0 15*
 -8 60 -63
 -6 145-147
 -4 0 -5*
 -2 149 152
  0 133 148
  2 0 -0*
  4 94 -91
  6 53 -53

H,K= 6, 9
 L FOB FCA
 -9 20 -29
 -7 27 -14
 -5 56 53
 -3 65 60
 -1 66 71
  1 64 65
  3 0 -8*
  5 51 -46

H,K= 6, 10
 L FOB FCA
 -6 51 44
 -4 75 63
 -2 60 53
  2 79 -69
  0 0 -7*
  4 97 -76

H,K= 6, 11
 L FOB FCA
 -5 72 -70
 -3 95 -81
 -1 0 -3*
  1 85 83
  3 77 78
  5 0 -5*

H,K= 6, 12
 L FOB FCA
 -2 53 -49
  0 0 -4*
  2 73 62

H,K= 7, 1
 L FOB FCA
-12 19 -13
-10 118-112
 -8 174-170
 -6 14 15
 -4 147 136
 -2 79 73
  0 197 203
  2 68 -64
  4 27 -28
  6 0 19*

  8 45 -40
 10 82 -79

H,K= 7, 2
 L FOB FCA
-13 49 60
-11 73 64
 -9 17 -20
 -7 171-168
 -5 177-167
 -3 51 50
 -1 343 337
  1 180 182
  3 121-113
  5 100-102
  7 15 -22
  9 0 24*
 11 42 -43

H,K= 7, 3
 L FOB FCA
-12 19 -8
-10 128 127
 -8 197 182*
 -6 20 5
 -4 18 -15
 -2 49 -48
  0 138-145
  2 70 -64
  4 76 -67
  6 21 28
  8 108 99
 10 114 105

H,K= 7, 4
 L FOB FCA
-11 68 70
 -9 71 68
 -7 52 -49
 -5 115-115
 -3 89 -91
 -1 90 -87
  1 12 18
  3 117 119
  5 67 64
  7 78 76
  9 25 7

H,K= 7, 5
 L FOB FCA
-12 35 -28
-10 19 24
 -8 38 39
 -6 38 -40
 -4 215-199
 -2 222-214
  0 44 21
  2 129 123
  4 120 119
  6 0 -3*
  8 94 -92

H,K= 7, 6
 L FOB FCA
-11 119-123
 -9 99-101
 -7 38 40
 -5 221 220
 -3 200 198
 -1 102-105
  1 141-140
  3 110-108
  5 59 -56
  7 34 -33
  9 18 -14

H,K= 7, 7
 L FOB FCA
-10 44 45
 -8 18 -16
 -6 0 -9*
 -4 75 73
 -2 158 158
  0 36 32
  2 67 -58
  4 108 -94
  6 38 31
  8 87 76

H,K= 7, 8
 L FOB FCA
 -9 76 76
 -7 122 119
 -5 17 24
 -3 74 -83
 -1 73 -72
  1 49 40
  3 88 87
  5 43 36
  7 0 5*

H,K= 7, 9
 L FOB FCA
 -8 76 -74
 -6 0 5*
 -4 47 51
 -2 0 4*
  0 38 -45
  2 35 -44
  4 25 20
  6 0 -13*

H,K= 7, 10
 L FOB FCA
 -7 98 -90
 -5 127-125
 -3 26 21
 -1 68 59
  1 18 -4
  3 67 -59
  5 27 -19

H,K= 7, 11
 L FOB FCA
 -6 0 6*
 -4 0 -4*
 -2 61 68
  0 83 89
  2 39 40
  4 34 -28

H,K= 7, 12
 L FOB FCA
 -3 35 19
 -1 35 32
  1 35 36

H,K= 8, 0
 L FOB FCA
 -2 131-129
  0 129-136
  2 421 380
  4 292 264
  6 47 40
  8 45 -34
 10 25 -18

H,K= 8, 1
 L FOB FCA
-13 83 79
-11 26 -15
 -9 91 -79
 -7 49 -46
 -5 57 -51
 -3 225-205
 -1 268-273
  1 198-190
  3 255 243
  5 337 306
  7 137 128
  9 119-113
 11 187-176

H,K= 8, 2
 L FOB FCA
-12 19 -3
-10 71 -72

 -8 152-137
 -6 244-228
 -4 83 -80
 -2 20 48
  0 179 191
  2 63 -51
  4 190-180
  6 160-154
  8 91 -92
 10 67 -69

H,K= 8, 3
 L FOB FCA
-11 32 26
 -9 51 44
 -7 69 73
 -5 24 30
 -3 31 30
 -1 0 3*
  1 197-187
  3 40 45
  5 76 77
  7 58 47
  9 24 0

H,K= 8, 4
 L FOB FCA
-12 0 23*
-10 26 15
 -8 78 73
 -6 186 178
 -4 149 144
 -2 83 75
  0 180-191
  2 153-147
  4 78 65
  6 246 237
  8 188 179
 10 55 56

H,K= 8, 5
 L FOB FCA
-11 48 -38
 -9 31 -46
 -7 33 -22*
 -5 50 46
 -3 42 34
 -1 19 10
  1 27 -27
  3 35 -40
  5 22 -17
  7 24 -11
  9 18 5

H,K= 8, 6
 L FOB FCA
-10 14 7
 -8 18 -6
 -6 46 -47
 -4 71 -58
 -2 29 25
  0 48 -52
  2 152-140
  4 158-148
  6 29 -35
  8 80 75

H,K= 8, 7
 L FOB FCA
 -9 27 14
 -7 41 -40
 -5 75 -75
 -3 0 4*
 -1 164 172
  1 104 106*
  3 16 -24
  5 174-163
  7 56 -53

H,K= 8, 8
 L FOB FCA
-10 20 -28
 -8 33 -31
 -6 18 21
 -4 0 1*
 -2 0 -7*
  0 122 131
  4 91 73
  6 64 -66

H,K= 8, 9
 L FOB FCA
 -9 35 -30
 -7 27 31
 -5 41 37
 -3 40 32
 -1 0 -8*
  1 35 -41
  3 18 -15
  5 0 8*
  7 48 -47

H,K= 8, 10
 L FOB FCA
 -6 0 1*
 -4 78 -79
 -2 78 -78
  0 19 -2
  2 37 41
  4 42 33
  6 0 -23*

H,K= 8, 11
 L FOB FCA
 -5 0 -10*
 -3 55 -52
 -1 55 -55
  1 33 -38
  3 62 61
  5 143 127

H,K= 8, 12
 L FOB FCA
 -2 56 49
  0 20 17
  2 54 -57

H,K= 9, 1
 L FOB FCA
-12 39 48
-10 43 43
 -8 66 -62
 -6 139-138
 -4 122-118
 -2 16 -9
  0 198 201
  2 26 24
  4 0 -38*
  6 38 -35
  8 0 -4*
 10 36 -28

H,K= 9, 2
 L FOB FCA
-13 68 -64
-11 19 -9
 -9 106 98
 -7 195 187
 -5 142 130
 -3 138-125
 -1 225-245*
  1 37 29
  3 41 32
  5 0 2*
  7 22 30
  9 42 47

H,K= 9, 3
 L FOB FCA
-12 20 -3
-10 18 -6
 -8 33 25
 -6 167 164
 -4 132 125
 -2 107-107
  0 147-151
  2 72 -75

  6 111 110
  8 83 84
 10 32 37

H,K= 9, 4
 L FOB FCA
-11 54 39
 -9 50 48
 -7 87 -79
 -5 181-164
 -3 194-180
 -1 0 -7*
  1 75 80
  3 119 112
  5 36 48
  7 46 -45
  9 36 -30

H,K= 9, 5
 L FOB FCA
-12 102-107
-10 114-105
 -8 17 7
 -6 86 84
 -4 86 85
 -2 34 37
  0 28 -31
  2 0 9*
  4 21 -28
  6 126-125
  8 126-119

H,K= 9, 6
 L FOB FCA
-11 28 -31
 -9 114-110
 -7 0 -13*
 -5 191 179
 -3 297 268
 -1 86 96
  1 107-115
  3 200-181
  5 36 38
  7 55 55

H,K= 9, 7
 L FOB FCA
-10 84 85
 -8 26 19
 -6 0 -24*
 -4 23 -9
 -2 0 3*
  0 0 9*
  2 55 -64
  4 0 -11*
  6 109 99
  8 86 74

H,K= 9, 8
 L FOB FCA
 -9 20 -15
 -7 87 -87
 -5 104 -96
 -3 34 -37
 -1 47 48
  1 50 53
  3 0 -20*
  5 82 -74
  7 56 -54

H,K= 9, 9
 L FOB FCA
 -8 28 -18
 -6 113 -98
 -4 137-131
 -2 0 -18*
  0 80 81
  2 99 90
  4 18 -15
  6 71 -67
  8 20 -36

H,K= 9, 10
 L FOB FCA
 -7 76 60
 -5 72 69
 -3 19 22
 -1 69 -70
  1 18 -5
  3 73 66
  5 115 103
  7 50 48

H,K= 9, 11
 L FOB FCA
 -4 87 78
 -2 28 36
  0 0 -6*
  2 20 -7
  4 0 5*

H,K= 9, 12
 L FOB FCA
 -1 35 -39*
  1 0 23*

H,K= 10, 0
 L FOB FCA
 -4 199-190
 -2 320-326
  0 395-426
  2 137 126
  4 331 302
  6 208 199
  8 37 37
 10 92 -88

H,K= 10, 1
 L FOB FCA
-13 41 -26
-11 32 -24
 -9 56 -58
 -7 112-112
 -5 96 -94
 -3 43 -36
 -1 76 -71
  1 92 -82
  3 33 -29
  5 66 -67
  7 0 4*
  9 30 -54

H,K= 10, 2
 L FOB FCA
-12 36 29
-10 72 -70
 -8 101-103
 -6 26 21
 -4 147 187
 -2 182 183
  0 48 47
  2 57 -59
  4 57 -61
  6 40 35
  8 41 37
 10 18 -13

H,K= 10, 3
 L FOB FCA
-11 78 72
 -9 74 67
 -7 55 51
 -5 174 170
 -3 269 241
 -1 130 125
  1 93 -96
  3 210-206
  5 15 -21
  7 199 188
  9 236 220

H,K= 10, 4
 L FOB FCA
-12 75 -74
-10 32 31
 -8 66 63
 -6 0 -26*

 -6 128-117
 -2 67 -46
  0 109 115
  2 137 123
  4 162-143
  6 241-229
  8 77 -74

H,K= 10, 5
 L FOB FCA
-11 39 -29
 -9 63 -56
 -7 29 -27
 -5 27 -28
 -3 176-164
 -1 150-145
  1 32 -32
  3 59 63
  5 22 -32
  7 123-116
  9 127-118

H,K= 10, 6
 L FOB FCA
-10 19 10
 -8 0 -17*
 -6 50 -55
 -4 47 41
 -2 119 118
  0 52 53
  2 70 -70
  4 51 -44
  6 38 33*
  8 32 31

H,K= 10, 7
 L FOB FCA
 -9 0 9*
 -7 0 -4*
 -5 34 -37
 -3 0 -6*
 -1 50 54
  1 149 160
  3 235 222
  5 91 89
  7 61 -49
  9 85 -76

H,K= 10, 8
 L FOB FCA
 -8 0 4*
 -6 32 29
 -4 0 5*
 -2 34 -34
  0 53 -60
  2 0 -9*
  4 70 67
  6 32 36
  8 0 -14*

H,K= 10, 9
 L FOB FCA
 -7 20 15
 -5 19 -5
 -3 0 -5*
 -1 0 -17*
  1 69 -76
  3 99 -94
  5 42 -47
  7 34 24

H,K= 10, 10
 L FOB FCA
 -6 0 1*
 -4 19 -13
 -2 73 -58
  0 79 -77
  2 0 1*
  4 67 62
  6 102 92

H,K= 10, 11
 L FOB FCA
 -5 20 24
 -3 49 47
 -1 0 17*
  1 48 -50
  3 28 -35

H,K= 11, 1
 L FOB FCA
-12 56 -44
-10 36 27
 -8 142 143
 -6 103 98
 -4 46 -42
 -2 103 -99
  0 48 -53
  2 0 21*
  4 85 84
  6 0 -15*
  8 62 -55
 10 41 36

H,K= 11, 2
 L FOB FCA
-11 91 -87
 -9 17 -15
 -7 157 150
 -5 263 253
 -3 99 96
 -1 259-257
  1 212-219
  3 117-115
  5 86 81
  7 85 81
  9 18 16

H,K= 11, 3
 L FOB FCA
-12 63 55
-10 18 -15
 -8 109-104
 -6 102-102
 -4 99 -96
 -2 0 11*
  0 18 19
  2 44 -46
  4 0 -8*
  6 63 62
  8 42 45
 10 53 -33

H,K= 11, 4
 L FOB FCA
-11 19 -11
 -9 70 -68
 -7 107-104
 -5 71 -59
 -3 113 108
 -1 76 73
  1 48 53
  3 0 -13*
  5 69 -69
  7 56 -62
  9 48 -40

H,K= 11, 5
 L FOB FCA
-10 63 -60
 -8 88 -78
 -6 0 -3*
 -4 182 173
 -2 234 225
  0 170 176
  2 64 -69
  4 142-139
  6 88 -84
  8 18 2
 10 27 32

H,K= 11, 6
 L FOB FCA
-11 61 48
 -9 113 110
 -7 79 78
 -5 64 -63

 -3 103-102
 -1 15 -37
  1 43 53
  3 125 117
  5 92 77
  7 18 -3
  9 27 21

H,K= 11, 7
 L FOB FCA
-10 20 -20
 -8 19 -11
 -6 31 27
 -4 48 -49
 -2 115-112
  0 132-134
  2 0 -4*
  4 103 97
  6 76 51
  8 0 -10*

H,K= 11, 8
 L FOB FCA
 -9 40 -28
 -7 148-133
 -5 120-109
 -3 0 10*
 -1 61 68
  1 45 56
  3 49 -60
  5 68 -62*
  7 27 -16
  9 20 9

H,K= 11, 9
 L FOB FCA
 -8 121 117
 -6 61 59
 -4 41 -49
 -2 74 -78
  0 0 4*
  2 83 74
  4 46 48
  6 27 3

H,K= 11, 10
 L FOB FCA
 -5 136 127
 -3 27 22
 -1 62 -62
  1 46 -46
  3 61 56
  5 71 62

H,K= 11, 11
 L FOB FCA
 -4 20 -25
 -2 28 41
  0 20 -9
  4 45 -45
  2 56 -59

H,K= 12, 0
 L FOB FCA
 -4 221-221
 -2 97 -97
  0 87 62
  2 52 -47
  4 233-223
  6 22 -14
  8 143 141
 10 100 96

H,K= 12, 1
 L FOB FCA
-11 57 -46
 -9 25 -20
 -7 16 -20
 -5 0 -20*
 -3 13 16
 -1 179 182
  1 196 201
  3 161 159
  5 69 -68
  7 227-219
  9 73 -72
 11 39 41

H,K= 12, 2
 L FOB FCA
-12 35 -26
-10 78 -80
 -8 17 14
 -6 209 203
 -4 222 201
 -2 128 123
  0 82 -84
  2 19 -27
  4 14 -14
  6 27 31
  8 38 37
 10 26 10

H,K= 12, 3
 L FOB FCA
-11 19 -9
 -9 18 -3
 -7 16 -4
 -5 56 -57
 -3 0 -6*
 -1 40 44
  1 0 -17*
  3 116-109
  5 117-104
  7 53 -58
  9 54 57
 11 81 81
 11 81 81

H,K= 12, 4
 L FOB FCA
-10 19 22
 -8 25 -15
 -6 107-102
 -4 130-122
 -2 111-111
  0 81 73
  2 100 98
  4 46 57
  6 40 -38
  8 144-143
 10 79 -83

H,K= 12, 5
 L FOB FCA
-11 35 27
 -9 0 10*
 -7 39 36
 -5 62 57
 -3 0 18*
 -1 49 -50
  1 45 -44
  3 86 81
  5 83 83
  7 30 30
  9 38 -39
 11 46 -40
 11 46 -40

H,K= 12, 6
 L FOB FCA
-10 20 -19
 -8 0 2*
 -6 39 52
 -4 28 -20
 -2 65 -75
  0 0 11*
  2 107 103
  4 170 160
  6 97 96
  8 26 -22
 10 67 -57

H,K= 12, 7
 L FOB FCA
 -9 39 -44
 -7 41 -38

 -5 0 -26*
 -3 0 -3*
 -1 0 -5*
  1 66 -69
  3 17 0
  5 77 75
  7 49 51
  9 20 -21

H,K= 12, 8
 L FOB FCA
 -8 28 29
 -6 42 -32
 -4 57 39
 -2 78 79
  0 30 -1
  2 148-134
  4 164-151
  6 68 -64
  8 66 62

H,K= 12, 9
 L FOB FCA
 -7 49 53
 -5 60 54
 -3 32 -31
 -1 41 -38
  1 18 -29
  3 19 -23
  5 0 2*
  7 40 48

H,K= 12, 10
 L FOB FCA
 -6 0 3*
 -4 0 5*
 -2 27 -23
  0 0 9*
  2 0 -15*
  4 20 -5
  6 29 28

H,K= 12, 11
 L FOB FCA
 -3 0 -7*
 -1 60 52*
  1 80 76
  3 20 18

H,K= 13, 1
 L FOB FCA
-12 49 -46
-10 64 -58
 -8 34 -26
 -6 123 109
 -4 187 186
 -2 35 12
  0 111-110
  2 119-120
  4 61 -65
  6 22 23
  8 49 45
 10 19 13

H,K= 13, 2
 L FOB FCA
-11 64 56
 -9 18 -24
 -7 112-106
 -5 86 -83
 -3 67 64
 -1 103 111
  3 106-100
  5 51 -51
  7 24 15
  9 31 19
 11 20 -4

H,K= 13, 3
 L FOB FCA
-12 29 26
-10 38 33
 -8 17 -2
 -6 97 -91
 -4 149-142
 -2 109-106
  0 42 43
  2 144 147
  4 149 140
  6 0 -10*
  8 47 -50
 10 57 -53

H,K= 13, 4
 L FOB FCA
-11 34 -31
 -9 107-103
 -7 72 -68
 -5 69 72
 -3 156 181
 -1 117 123
  1 96 99
  3 0 -8*
  5 0 -13*
  7 17 -11
  9 26 -11
 11 29 -15

H,K= 13, 5
 L FOB FCA
-10 91 92
 -8 68 66
 -6 34 -36
 -4 89 -91
 -2 22 28
  0 132 131
  2 66 67
  4 101 -92
  6 106-104
  8 41 28
 10 91 91

H,K= 13, 6
 L FOB FCA
 -9 95 86
 -7 143 141
 -5 17 1
 -3 260-254
 -1 257-265
  1 42 -46
  3 57 56
  5 42 44
  7 26 -22
  9 0 -16*

H,K= 13, 7
 L FOB FCA
 -8 43 -46
 -6 31 -28
 -4 0 16*
 -2 29 -25
  0 47 -52
  2 0 -13*
  4 30 27
  6 61 59
  8 20 -12

H,K= 13, 8
 L FOB FCA
 -7 27 25
 -5 78 71
 -3 56 56
 -1 0 13*
  1 0 -17*
  3 0 11*
  5 62 61
  7 39 47

H,K= 13, 9
 L FOB FCA
 -6 48 47
 -4 98 93
 -2 58 57
  0 0 1*
  2 69 -63
  4 57 -51
  6 28 32

H,K= 13, 10
 L FOB FCA
 -5 98-100
 -3 0 -15*
 -1 47 49
  1 27 33
  3 48 -42
  5 70 -71

H,K= 13, 11
 L FOB FCA
 -2 29 -36
  0 54 -73
  2 0 -6*

H,K= 14, 0
 L FOB FCA
 -6 27 -31
 -4 46 -47
 -2 101 103
  0 275 263
  2 169 172
  4 0 4*
  6 129-123
  8 114-107
 10 0 2*

H,K= 14, 1
 L FOB FCA
-11 0 -20*
 -9 31 -17
 -7 64 56
 -5 85 91
 -3 70 69
 -1 53 60
  1 230 235
  3 188 181
  5 0 -3*
  7 132-124
  9 55 -42
 11 59 56

H,K= 14, 2
 L FOB FCA
-12 81 -81
-10 27 -18
 -8 91 96
 -6 85 80
 -4 21 -18
 -2 110-111
  0 104 -94
  2 20 -12
  4 0 -4*
  6 49 -45
  8 40 -33
 10 19 19

H,K= 14, 3
 L FOB FCA
-11 20 -10
 -9 82 -76
 -7 88 -81
 -5 0 4*
 -3 73 -71
 -1 136-144
  1 156-156
  3 79 80
  5 111 108
  7 0 4*
  9 128-116
 11 123-125

H,K= 14, 4
 L FOB FCA
-10 43 40
 -8 31 -36
 -6 73 -73
 -4 16 23
 -2 103 107
  0 56 -62
  2 80 -85
  4 32 30
  6 135 128
  8 128 114
 10 0 -20*

H,K= 14, 5
 L FOB FCA
 -9 114 106
 -7 47 54
 -5 24 -18
 -3 23 18
 -1 16 22
  1 58 72
  3 0 28*
  5 17 -24
  7 18 12
  9 87 84

H,K= 14, 6
 L FOB FCA
-10 0 -17*
 -8 19 -15
 -6 25 -26
 -4 26 -17
 -2 61 -56
  0 49 -34
  2 16 4
  4 0 5*
  6 26 11
  8 19 13

H,K= 14, 7
 L FOB FCA
 -9 35 -33
 -7 38 -30
 -5 18 6
 -3 52 58
 -1 52 51
  1 42 -53
  3 140-133
  5 123-111
  7 19 18
  9 46 42

H,K= 14, 8
 L FOB FCA
 -8 28 27
 -6 69 64
 -4 49 50
 -2 40 39
  0 0 -3*
  2 0 -6*
  4 32 -24
  6 47 -45
  8 20 -18

H,K= 14, 9
 L FOB FCA
 -7 35 26
 -5 0 -0*
 -3 42 -43
 -1 37 -31
  1 0 10*
  3 50 51
  5 59 54

H,K= 14, 10
 L FOB FCA
 -4 56 -54
 -2 0 3*
  0 83 81
  2 68 64
  4 0 -4*

H,K= 14, 11
 L FOB FCA
 -1 0 5*
  1 0 23*

H,K= 15, 1
 L FOB FCA
-10 0 -0*
 -8 39 -40
 -6 76 -68
 -4 15 -11

 -2 92 94
  0 115 121
  2 35 -48
  4 133-123
  6 49 -53*
  8 0 24*
 10 39 39

H,K= 15, 2
 L FOB FCA
-11 63 56
 -9 86 84
 -7 17 11
 -5 211-203
 -3 242-231
 -1 48 -44
  1 121 127
  3 176 171
  5 23 13
  7 39 -39
  9 37 -24
 11 0 8*

H,K= 15, 3
 L FOB FCA
-10 43 -30
 -8 18 14
 -6 72 64
 -4 16 -18
 -2 105-106
  0 30 30
  2 120 116
  4 139 134
  6 34 34
  8 77 -77
 10 59 -56

H,K= 15, 4
 L FOB FCA
-11 35 -26
 -9 38 -29
 -7 18 17
 -5 52 54
 -3 83 77
 -1 80 80
  1 15 -29
  3 0 -30*
  5 0 7*
  7 25 -35
  9 27 -31

H,K= 15, 5
 L FOB FCA
-10 20 -2
 -8 65 62
 -6 102 99
 -4 0 2*
 -2 80 -76
  0 220-230
  2 145-150
  4 17 -5
  6 62 69
  8 83 78
 10 29 22

H,K= 15, 6
 L FOB FCA
 -9 0 -5*
 -7 37 -29
 -5 0 3*
 -3 0 14*
 -1 41 -38
  1 63 -81
  3 67 -59
  5 25 15
  7 63 69
  9 20 23

H,K= 15, 7
 L FOB FCA
 -8 28 24
 -6 19 -7
 -4 36 -25
 -2 0 13*
  0 123 125
  2 104 105
  4 63 66
  6 0 -14*
  8 60 -61

H,K= 15, 8
 L FOB FCA
 -7 40 42
 -5 111 102
 -3 95 86
 -1 0 -6*
  1 36 -22
  3 0 0*
  5 0 -8*
  7 0 -3*

H,K= 15, 9
 L FOB FCA
 -6 92 -83
 -4 0 -14*
 -2 107 106
  0 38 36
  2 101 -94
  4 131-126
  6 45 -48

H,K= 15, 10
 L FOB FCA
 -3 96 -83
 -1 20 -0
  1 59 62
  3 28 22

H,K= 16, 0
 L FOB FCA
 -6 73 -25
 -4 111 106
 -2 122 120
  0 137 126
  2 74 75
  4 148 141
  6 58 60
  8 0 -3*
 10 112-100

H,K= 16, 1
 L FOB FCA
-11 44 41
 -9 26 18
 -7 54 -50
 -5 0 -16*
 -3 64 60
 -1 84 81
  1 33 -28
  3 224-208
  5 123-117
  7 60 52*
  9 108 98
 11 50 29*

H,K= 16, 2
 L FOB FCA
-10 19 20
 -8 25 18
 -6 24 18
 -4 70 -68
 -2 182-182
  0 126-120
  2 0 -8*
  4 36 -48
  6 74 -27
  8 18 -19
 10 0 -11*

H,K= 16, 3
 L FOB FCA
-11 0 -18
 -9 19 -9
 -7 18 22
 -5 44 51
 -3 61 -57
 -1 101 -97

  1 48 -54
  3 65 68
  5 170 157
  7 107 100
  9 27 -28

H,K= 16, 4
 L FOB FCA
-10 34 35
 -8 41 35
 -6 0 13*
 -4 73 61*
 -2 88 89
  0 87 93
  2 16 25
  4 67 -60
  6 0 1*
  8 82 83
 10 78 75

H,K= 16, 5
 L FOB FCA
 -9 20 -12
 -7 26 -29
 -5 35 -36
 -3 0 -14*
 -1 43 48
  1 28 29
  3 0 4*
  5 56 -64
  7 46 -35
  9 34 28

H,K= 16, 6
 L FOB FCA
 -8 19 -13
 -6 41 -28
 -4 70 -32
 -2 0 -4*
  0 29 18
  2 34 -34
  4 75 -73
  6 49 -45
  8 44 -47

H,K= 16, 7
 L FOB FCA
 -7 87 63
 -5 79 73
 -3 18 7
 -1 25 -26
  1 47 42
  3 28 19
  5 32 -43
  7 106-100

H,K= 16, 8
 L FOB FCA
 -6 39 39
 -4 19 10
 -2 26 -34
  0 26 -29
  2 19 17
  4 66 59
  6 69 62

H,K= 16, 9
 L FOB FCA
 -5 85 -80
 -3 0 -19*
 -1 0 27*
  1 0 5*
  3 0 18*
  5 0 17*

H,K= 16, 10
 L FOB FCA
 -2 0 -1*
  0 0 4*
  2 28 21

H,K= 17, 1
 L FOB FCA
-10 41 34
 -8 79 65
 -6 0 -13*
 -4 105-103
 -2 123-114
  0 62 -54
  2 120 116
  4 97 93
  6 17 -21
  8 32 -14
 10 0 4*

H,K= 17, 2
 L FOB FCA
-11 20 -12
 -9 33 34
 -7 50 51
 -5 40 37
 -3 31 -32
 -1 75 -84
  1 56 -59
  3 115 117
  5 120 115
  7 40 40
  9 19 -16

H,K= 17, 3
 L FOB FCA
-10 52 -46
 -8 64 -57
 -6 24 -14
 -4 80 78
 -2 149 146
  0 98 100
  2 29 -28
  4 69 -72
  6 53 -58
  8 0 -6*
 10 29 20

H,K= 17, 4
 L FOB FCA
 -9 0 1*
 -7 79 76
 -5 108 91
 -3 0 -6*
 -1 131-128
  1 83 -81
  3 60 -53
  5 17 -24
  7 26 -20
  9 20 -15

H,K= 17, 5
 L FOB FCA
 -8 33 -25
 -6 48 43
 -4 90 82
 -2 74 74
  0 140-135
  2 176-185
  4 34 -47
  6 110 111
  8 76 74

H,K= 17, 6
 L FOB FCA
 -9 0 15*
 -7 119-109
 -5 128-118
 -3 0 -10*
 -1 174 189
  1 148 148
  3 40 24
  5 49 -51
  7 0 -18*
```

TABLE I (*Continued*)

```
H,K= 17, 7
 L FOB FCA
-8  29  34
-6   0 -10*
-4  78 -75
-2   0  -1*
 0 101  95
 2 104 113
 4   0  -5*
 6 101 -97

H,K= 17, 8
 L FOB FCA
-7   0  -9*
-5   0  -2*
-3   0   2*
-1  19 -16
 1  19 -14
 3  33 -38
 5  20 -18

H,K= 17, 9
 L FOB FCA
-4   0  -2*
-2  80 -72
 0  48 -53
 2  48  59

H,K= 17, 10
 L FOB FCA
-1   0  -6*
 1  20 -22

H,K= 17, 9
 L FOB FCA
 4  72  62

H,K= 18, 0
 L FOB FCA
-8  82 -73
-6  49 -51
-4  71  70
-2  89  96
 0  60  40
 2 147-147
 4 106-102
 6  39  32
 8  19   6
10  20 -30

H,K= 18, 1
 L FOB FCA
-9  19 -15
-7   0  -4*
-5  58  57
-3  98  96
-1  32 -36
 1 189-191
 3 240-229
 5 114-108
 7  18  16
 9  75  68

H,K= 18, 2
 L FOB FCA
-10  93  89
-8  95  73
-6  29  29
-4  82 -84
-2  73 -72
 0  22  15
 2  48  59
 4  60  56
 6  31  32
 8  57  58
10  54  52
10  54  52

H,K= 18, 3
 L FOB FCA
-9  97  89
-7  66  56
-5   0 -22*
-1  23  19
-3  37 -42
 1 176 182
 3 110 107
 5  43 -42
 7  52 -51
 9  28  25

H,K= 18, 4
 L FOB FCA
-10 115-104
-8  79 -71
-6  36  20
-4  62  63
-2  33 -31
 0  17 -25
 2   0 -27*
 4  17  -9
 6  52 -49
 8  91 -92

H,K= 18, 5
 L FOB FCA
-9  72 -58
-7  87 -81
-5  48 -43
-3  46  44
-1   0   3*
 1  78 -76
 3  63 -69
 5  18   1
 7  19  11

H,K= 18, 6
 L FOB FCA
-8  28  32
-6  27  31
-4  18   6
-2  47  42
 0  75  65
 2  62  69
 4  26  29
 6  33 -38
 8  29 -34

H,K= 18, 7
 L FOB FCA
-7  49  48
-5  27  22
-3   0   2*
-1   0  12*
 1  18  26
 3  37  32
 5  61  56*
 7  20  32

H,K= 18, 8
 L FOB FCA
-6  53 -60
-4  67 -64
-2  19 -27
 0  42 -40
 2  91 -95
 4   0  -2*

H,K= 18, 9
 L FOB FCA
-3  20 -23
-1  39 -54
 1  20  16
 3  28  36

H,K= 19, 1
 L FOB FCA
-10  20 -16
-8  19  28
-6  66  64
-4  24  24
-2  78 -75
 0 139-131
 2   0   4*
 4  48  48
 6  98  91
 8  19  24

H,K= 19, 2
 L FOB FCA
-9  97 -91
-7  98 -80
-5   0  17*
-3 139 134
-1 104  99
 1  57 -54
 3  80 -82
 5  25 -22
 7  46  47
 9  40  42

H,K= 19, 3
 L FOB FCA
-10   0 -12*
-8  19  -8
-6  54 -51
-4   0 -14*
-2 100  98
 0 102  90
 2   0  -5*
 4 124-116
 6 131-118
 8  19 -10

H,K= 19, 4
 L FOB FCA
-9  40  26
-7  70  58
-5  54  48
-1  51 -54
 1  66 -62
 3  46 -40
 5  32 -26
 7  19  -7
 9   0  26*

H,K= 19, 5
 L FOB FCA
-8  20  -3
-6  42 -30
-4  54 -43
-2  25 -26
 0  82  78
 2 107 105
 4  63  53
 6  27  20
 8  20 -16

H,K= 19, 6
 L FOB FCA
-7  20 -13
-5  57 -51
-3  32 -20
-1  11  38
 1  70  62
 3 103  88
 5  35  33
 7  20 -11

H,K= 19, 7
 L FOB FCA
-6  28 -35
-4  19  15
-2  42  38
 0   0  -9*
 2  78 -75
 4   0 -64*
 6  20 -18

H,K= 19, 8
 L FOB FCA
-5   0 -39*
-3 117-115
-1  39 -46
 1  19  21
 3  59  59
 5   0 -12*

H,K= 19, 9
 L FOB FCA
-2  57 -54
 0  72 -56
 2   0  -3*

H,K= 20, 0
 L FOB FCA
-8  42  33
-6 140 141
-4  17  12
-2  95 -93
 0  84 -81
 2  55 -50
 4  72 -62
 6 152-138
 8  89 -85

H,K= 20, 1
 L FOB FCA
-9  14  17
-7 105 107
-3  34  24*
-1 108-106
 1  50 -54
 3  17   7
 5  91  99
 7  63  61
 9  28 -20

H,K= 20, 2
 L FOB FCA
-8  19   6
-6  79 -70
-4  39 -43
-2  56  52
 0 118 115
 2  59  50
 4  30  32
 6  65  66
 8  62  64

H,K= 20, 3
 L FOB FCA
-9  45 -37
-7  46 -30
-5  25 -30
-3  30 -36
-1  29  41
 1  90  79
 3  17  20
 5  45 -40
 7  92 -92

H,K= 20, 4
 L FOB FCA
-8  62 -50
-6   0  24*
-4  31  18
-2  17 -15
 0  97 -92
 2  47 -59
 4  31  27
 6  19  18
 8  40 -40

H,K= 20, 5
 L FOB FCA
-7   0  19*
-5  19  17
-3   0  26*
-1   0  -6*
 1  18  -1
 3   0   6*
 5  46  50
 7  28  19

H,K= 20, 6
 L FOB FCA
-6   0  -9*
-4  38  36
-2  41  31
 0  55  60
 2   0  15*
 4   0 -11*
 6  34   9*

H,K= 20, 7
 L FOB FCA
-5  44 -40
-3  27 -30
-1  19 -20
 3  70 -69
 5   0 -24*

H,K= 20, 8
 L FOB FCA
-4  53 -44
-2  81 -71
 0  70  -6
 2  40  37
 4   0  -6*

H,K= 20, 9
 L FOB FCA
-1  41  47
 1  35  45

H,K= 21, 1
 L FOB FCA
-8  47 -38
-6  32 -17
-4  35  27*
-2  66  56
 0  59  49
 2   0  -9*
 4  59 -56
 6   0 -12*
 8  20  29*

H,K= 21, 2
 L FOB FCA
-9  20 -26
-7  46 -42
-5  51 -46
-3   0  -9*
-1  82  82
 1 134 125
 3  18  30
 5  84 -83
 7 115-115

H,K= 21, 3
 L FOB FCA
-8  28  14
-6  53  43
-4   0  30*
-2  35 -38
 0  78 -66
 2   0 -21*
 4  18 -15
 6   0  10*
 8   0 -17*

H,K= 21, 4
 L FOB FCA
-7  28  25
-5  59 -64
-3  36 -57*
-1  31  22
 1   0  19*
 3  16  24
 5   0   4*
 7  14  29

H,K= 21, 5
 L FOB FCA
-8  46  40
-6  19  -5
-4 116-100*
-2 117-114
 0   0   8*
 2 135 134
 4 138 142
 6  20  18

H,K= 21, 6
 L FOB FCA
-7  35 -26
-5  81  76
-3  76  74
-1  59 -64
 1  96 -87
 3  38 -41
 5  60  78

H,K= 21, 7
 L FOB FCA
-4  48  42
-2  67  57
 0  19  -1
 2  94 -92
 4  94 -92

H,K= 21, 8
 L FOB FCA
-3   0 -15*
-1   0  10*
 1   0  15*
 3   0  -3*

H,K= 22, 0
 L FOB FCA
-8 100 102
-6 102  85
-4  47  38
-2   0  -1*
 0  25  -8
 2   0  16*
 4   0   8*
 6  47 -47
 8   0 -19*

H,K= 22, 1
 L FOB FCA
-9  20  19
-7  27 -12
-1  25 -22
-3  18 -13
-5   0  -9*
 1  35 -36
 3  69  67
 5 119 101
 7  88  75

H,K= 22, 2
 L FOB FCA
-8 101 -94
-6  71 -67
-4  26  29
-2   0   9*
 0  18 -21
 2  40 -24
 4  18   8
 6  38  30
 8  20 -14

H,K= 22, 3
 L FOB FCA
-7  83 -66
-5  65 -62
-3  44  45
-1  56  67
 1   0  -1*
 3  61 -56
 5  35 -45
 7  20 -18

H,K= 22, 4
 L FOB FCA
-9  98  84
-6  19  20
-4  72 -59
-2  41 -35
 0  51  47
 2  71  75
 4   0   3*
 6  30 -36

H,K= 22, 5
 L FOB FCA
-7  45  41
-5  91  41
-3  19   4
-1  19  -2
 1  19   1
 3   0  12*
 5   0  10*

H,K= 22, 6
 L FOB FCA
-6   0 -11*
-4   0   4*
-2   0  19*
 0  51 -47
 2  77 -76
 4  40 -31

H,K= 22, 7
 L FOB FCA
-3  45 -49
-1  56 -51
 1   0  14*
 3  20  17

H,K= 22, 8
 L FOB FCA
-2   0   6*
 0  29  41

H,K= 23, 1
 L FOB FCA
-8  21  -4
-6  47 -34
-4  78 -77
-2   0   5*
 0  91  80
 2  79  64
 4   0  -9*
 6  65 -58

H,K= 23, 2
 L FOB FCA
-7  62  47
-5  60  56
-3  41 -39
-1  82 -50
 1  18 -13
 3  52  50
 5  38  31
 7  35 -38

H,K= 23, 3
 L FOB FCA
-6  75  69
-4  86  71
-2   0   3*
 0 102 -99
 2  83 -75
 4   0  10*
 6  77  61

H,K= 23, 4
 L FOB FCA
-7  29 -30
-5  64 -59
-3  33 -33
-1   0  -4*
 1   0  -4*
 3  43  37
 5  48  52

H,K= 23, 5
 L FOB FCA
-6  45 -37
-4   0 -12*
-2   0   3*
 0   0  -7*
 2  19  -0
 4   0   2*

H,K= 23, 6
 L FOB FCA
-5  35  31
-3  52  38
-1  62  53
 1   0 -14*
 5  40 -57

H,K= 23, 7
 L FOB FCA
-2   0  12*
 0   0 -14*
 2   0  -7*

H,K= 24, 0
 L FOB FCA
-6  34 -23
-4  19   9
-2  18  12
 0  26  30
 2   0 -11*
 4  27 -24
 6  79  75

H,K= 24, 1
 L FOB FCA
-7  40  39
-5  77 -61
-3 137-135
-1  69 -59
 1  26  25
 3  86  77
 5  28  15
 7  20 -17

H,K= 24, 2
 L FOB FCA
-6  28   9
-4  27   8
-2  19 -29
 0  32 -20
 2  19 -12
 4  33  31
 6  35 -36

H,K= 24, 3
 L FOB FCA
-7  20   3
-5  19   0
-3  54  38
-1  62  51
 1  27  36
 3  63 -60
 5  59 -53

H,K= 24, 4
 L FOB FCA
-6  53  43
-4   0   3*
-2   0  -3*
 0  45  47
 2  33  25
 4   0 -12*

H,K= 24, 5
 L FOB FCA
-5  29  25
-3   0   2*
-1  27 -28
 1  19 -28
 3  20 -14

H,K= 24, 6
 L FOB FCA
-4  20 -32
-2  49 -45
 0  44 -39
 2  20 -25

H,K= 25, 1
 L FOB FCA
-6   0  -5*
-4   0   5*
-2  19 -21
 0  38 -22
 2  19  28
 4  58  40
 6  20  16

H,K= 25, 2
 L FOB FCA
-5  56  45
-3  54  47
-1   0  -6*
 1  60 -45
 3  91 -72
 5  20  -8

H,K= 25, 3
 L FOB FCA
-6  20  15
-4   0   5*
-2  38 -35
 0  47 -36
 2   0   4*
 4   0 -13*

H,K= 25, 4
 L FOB FCA
-5  24 -12
-3  28  -8
-1  19 -26
 1  34 -34
 3   0  12*

H,K= 25, 5
 L FOB FCA
-4  20  21
-2  82  67
 0 107  95
 2   0   3*

H,K= 25, 6
 L FOB FCA
-1   0  19*
 1  70  61

H,K= 26, 0
 L FOB FCA
-6  29 -45
-4 100 -98
-2  75 -50
 0  33 -26
 2   0  14*
 4  20   0

H,K= 26, 1
 L FOB FCA
-5  85 -87
-3  75 -64
-1  19 -11
 1  61  54
 3  48  41
 5  41 -27

H,K= 26, 2
 L FOB FCA
-4   0  19*
-2  39  43
 0  19  26
 2  28  19
 4   0  -7*

H,K= 26, 3
 L FOB FCA
-5  88  75
-3  66  63
-1  20 -13
 1  83 -86
 3  20  -3

H,K= 26, 4
 L FOB FCA
-4   0   7*
-2   0  11*
 0  49 -40
 2  40 -34

H,K= 26, 5
 L FOB FCA
-1  20  13
 1  35  30

H,K= 27, 1
 L FOB FCA
-4  49  35
-2  44  36
 0  28   7
 2  69 -58
 4  41 -31

H,K= 27, 2
 L FOB FCA
-3  20  -0
-1  64  27
 1  40  37
 3  45 -45

H,K= 27, 3
 L FOB FCA
-2  98 -81
 0  20   7
 2  61  49

H,K= 27, 4
 L FOB FCA
-1  50  39
 1  20  29

H,K= 28, 0
 L FOB FCA
-2  35 -37
 0  60 -56
 2  29  -6

H,K= 28, 1
 L FOB FCA
-3  81  70
-1 132 117
 1  70  63

H,K= 28, 2
 L FOB FCA
 0  20   8
-2   0   4*
```

[a] The signs of F_c apply to the real part of the structure factor. The asterisks indicate data not used in the refinement.

particular nuclear resonance depends upon a number of factors, among which is the average value of the so-called geometric factor. For a paramagnetic ion in an axially symmetric ligand field this is

$$\left\langle \frac{3 \cos^2 \chi - 1}{R^3} \right\rangle_{\text{av}}$$

where χ is the angle made by a vector from the transition metal atom to the nucleus in question with the principal axis and R is the length of this vector. For a given system at a particular temperature the different dipolar shifts experienced by nonequivalent nuclei result from their different geometric factors. Thus, in principle at least, it should be possible to obtain information about the geometry of ion pairing in solution from the observed dipolar shifts. Interionic distances may be estimated by comparing observed dipolar shift ratios with average values of geometric factor ratios calculated on the basis of various ion-pairing models as a function of interionic distance. The first such attempt was by LaMar, who estimated[3] a 3.8-Å (Co–N) separation between the counterions of $[(n\text{-}C_4H_9)_4N][Co(C_6H_5)_3PI_3]$ in chloroform solution. The model used involved no preferred rotational orientation of the cation as it approached the anion along the latter's C_3 axis on the side opposite the bulky triphenylphosphine ligand. Owing to the flexibility of the butyl chains of the cation, only the inner two sets of methylene protons were employed in the estimate. The use of the above "spherical" model for the tetra-*n*-butylammonium ion has been criticized.[7] A model based on rigidly extended butyl chains gives a more reasonable estimate[7] of this interionic distance (6 Å). Indeed, a recent crystal structure determination by Horrocks, *et al.*,[8] shows that the tetra-*n*-butylammonium ion approaches to within 6.0 Å of the $Ni(quin)Br_3^-$ (quin = quinoline) ion in the solid state. This anion has less bulk than $Co(C_6H_5)_3PI_3^-$. In a recent attempt[6] to remedy the uncertainties arising from the nonrigidity of the butyl chains, a proton magnetic resonance (pmr) study of the tetraphenylarsonium salt of $Co(C_6H_5)_3PI_3^-$ was undertaken. Again a model based on approach by the cation along the C_3 axis of the anion was employed and an interionic distance in solution of ~9.0 Å was obtained from the two independent dipolar shift ratios. While ion association in solution and solid-state structure are not strictly comparable, it was nevertheless of interest to determine the mode and closeness of counterion approach of a tetraphenylarsonium salt of a $M(C_6H_5)_3PI_3^-$ ion in the solid state. Furthermore, the crystal structures of only three tetrahedral or pseudotetrahedral complexes of nickel(II) involving monodentate ligands have been determined[8-10] and additional information on this relatively unusual class of coordination compounds is desirable.

Experimental Section

Dark red, platelike monoclinic crystals of tetraphenylarsonium triiodo(triphenylphosphine)nickelate(II), $[(C_6H_5)_4As][Ni(C_6H_5)_3PI_3]$, were prepared as described elsewhere.[6] Systematic absences on Weissenberg film layers *h*0*l* through *h*3*l* were: for *hkl*, $h + k + l = 2n + 1$ and for *h*0*l*, $l = 2n + 1$; these imply space groups Ic or I2/c. The density measured by flotation in methylene iodide–carbon tetrachloride was 1.73 ± 0.02 g/cm^3 which compares favorably with the calculated density of 1.745 g/cm^3 for $Z = 8$. With eight molecules per unit cell the space group I2/c, with an eightfold general position, was chosen and this choice was confirmed by the subsequent successful refinement of the structure. The nonstandard space group I2/c which is equivalent to C2/c (C_{2h}^6, no. 15) was chosen in order to obtain a β angle reasonably close to 90°. The general equivalent positions of I2/c are: x, y, z; $\frac{1}{2} + x, \frac{1}{2} + y, \frac{1}{2} + z$; $x, -y, \frac{1}{2} + z$; $\frac{1}{2} + x, \frac{1}{2} - y, z$; $-x, -y, -z$; $\frac{1}{2} - x, \frac{1}{2} - y, \frac{1}{2} - z$; $-x, y, \frac{1}{2} - z$; $\frac{1}{2} - x, \frac{1}{2} + y, -z$. A crystal of approximate dimensions 0.18 × 0.13 × 0.05 mm was chosen for intensity measurements and was mounted on a quartz fiber with the *b* axis (unique) of the monoclinic cell parallel to the spindle axis of the instrument. The unit cell dimensions were

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(7) D. W. Larsen, *Inorg. Chem.*, **5**, 1109 (1966).

(8) W. D. Horrocks, Jr., D. H. Templeton, and A. Zalkin, *ibid.*, **7**, 2303 (1968).

(9) P. Pauling, *ibid.*, **5**, 1948 (1966).

(10) G. Garton, D. E. Henn, H. M. Powell, and L. M. Venanzi, *J. Chem. Soc.*, 3625 (1963).

determined at room temperature on a General Electric XRD-5 unit equipped with a manual goniostat, scintillation counter, and pulse height discriminator using unfiltered molybdenum radiation (λ 0.70926 Å for Kα_1). They are a = 33.971 ± 0.004 Å, b = 14.992 ± 0.002 Å, c = 16.253 ± 0.004 Å, and β = 92.06 ± 0.05°. The standard deviations of the cell dimensions were determined from the variations in the values obtained from careful measurement of the h00, 0k0, and 00l reflections at high 2θ values where α_1, α_2 separation was achieved.

The intensities of 2548 independent reflections were measured using zirconium-filtered molybdenum radiation out to 2θ = 35° by 10-sec peak counts with a 4° takeoff angle. Standard reflections were measured approximately every 100 measurements and the intensity was maximized as a function of ϕ rotation at every new ϕ setting if a suitably strong reflection was available. No appreciable change (<15%) was noted in the intensities of the standard reflections throughout the course of the measurements. The intensities were corrected for background from a previously measured general background curve which showed the background to be a smoothly decreasing function of 2θ alone (not χ or ϕ). After this correction, 342 of the reflections were recorded as zero. The intensities were assigned standard deviations according to the formula (where I = counts per second)

$$\sigma(I) = [I + (0.05I)^2]^{1/2}$$

except that, when any net intensity was less than or equal to 2 counts/sec, it was assigned the standard deviation $\sqrt{2}$. Values for F_o were obtained from the intensities after correction[11] for Lp (Lorentz, polarization) and were assigned standard deviations as

$$\sigma(F_o) = \sigma(I)/2[(\mathrm{Lp})(I)]^{1/2}$$

The variation in the intensity of the 020 reflection as a function of ϕ rotation (at χ = 90°) is shown in Figure 1 and indicates that the data suffer to some extent from absorption errors. The linear absorption coefficient for molybdenum radiation is 36 cm^{-1}. The absorption was maximum at ϕ values of 79.09 and 259.09° corresponding to the $-a^*$ and a^* directions. The plate face of the crystal was parallel to the (100) plane. The b axis was parallel to the 0.13-mm dimension of the crystal. The structure was satisfactorily solved and refined (*vide infra*) without the use of absorption corrections and, since all of the chemically significant information was revealed without applying such corrections, none was made.

Solution and Refinement of the Structure

A three-dimensional Patterson synthesis[11] located the nickel, arsenic, and three iodine atoms. Three cycles of least-squares (LS) refinement[11] yielded a conventional R value[12] of 0.34. A Fourier synthesis[11] disclosed the phosphorus and all 42 carbon atoms. Further LS refinement with isotropic temperature factors lowered R to 0.12. At this point the six atoms heavier than carbon were allowed anisotropic temperature factors and the structure was further refined by three cycles of LS to a final R value of 0.082. The final refinement was carried out using 2180 nonzero data (26 nonzero data required remeasurement at the end and were not included in the refinement as indicated by the asterisks in Table I). A final difference Fourier revealed a residual electron density consistent with the hydrogen atoms in the structure which were not located. Atomic scattering factors for the neutral atoms and dispersion corrections for the neutral atoms were taken from the "International Tables."[13]

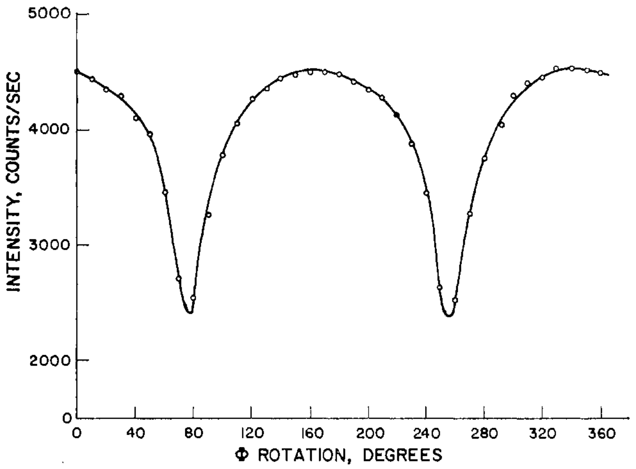

Figure 1.—Intensity *vs.* ϕ rotation for the 020 reflection.

Table I gives the final observed and calculated structure factors. On the final full-matrix LS cycle the largest variation of an atomic positional parameter was 0.0002 while the largest temperature factor shift was 0.05; in all cases the shifts were smaller than the estimated standard deviations. The error in an observation of unit weight at the end was 1.47. The final heavy-atom parameters are given in Table II.

Description and Discussion of the Structure

Phenyl Ring Parameters.—Although they are of no particular chemical interest, the structures of seven phenyl groups were determined in the course of this work. The carbon–carbon bond distances and angles are given in Table III. The average C–C bond distance is 1.407 Å, in good agreement with the accepted value for aromatic C–C bonds. The individual values range from 1.326 to 1.514 Å with a standard deviation for an individual measurement of 0.044 Å calculated from $\sigma = (\Sigma_i \Delta_i^2/(m - 1))^{1/2}$ for the 42 measurements. This is in good agreement with the esd's derived from the inverse matrix (Table III). Furthermore the deviations from the mean value conform to χ^2. The phenyl rings are quite accurately planar, with no carbon deviating by more than 0.06 Å from the best least-squares plane through the individual phenyl rings, and in most cases the planarity is considerably better than that. The 42 C–C–C bond angles (Table III) are, with six exceptions, within ±5° of the expected 120° angle. The mean value is 119.9° with an esd for an individual measurement of 4.0°, slightly higher than those derived from the inverse matrix. The structural deviations of the phenyl rings from ideality may be taken as some measure of the quality of the present determination, although the parameters of the heavier atoms are felt to be considerably more accurate than those of the carbon atoms.

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(11) Computations were performed on a CDC 6600 computer at LRL. The following computer programs were used for the operations indicated: instrumental setting, Zalkin's GONIO (a modification of a program written by D. W. Larsen); reduction of new data, Zalkin's INCORE; Patterson and Fourier syntheses, Zalkin's FORDAP; least squares refinement, Zalkin's LS 250 (a modification of the Gantzel–Sparks–Trueblood full-matrix program which minimizes $\Sigma w|\Delta F|^2/\Sigma w|F_o|^2$, $w = 1/\sigma^2(F)$); interatomic distance and angle computation, Zalkin's DISTAN; calculation of least-squares planes, Chu's LSPLAN; graphical representation of atomic positions and molecular stereochemistry, Zalkin's ATMPLT and Johnson's ORTEP; preparation of Table I, Zalkin's LIST.

(12) $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$.

(13) "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, p 201 ff.

TABLE II

Final Atomic Positional and Isotropic Thermal Parameters for $[(C_6H_5)_4As][Ni(C_6H_5)_3PI_3]$[a]

Atom[b]	x	y	z	B, Å^2
As	0.4176 (1)	0.2767 (2)	0.9009 (2)	...
I(1)	0.3423 (1)	0.1158 (1)	0.5990 (1)	...
I(2)	0.3674 (1)	0.3852 (1)	0.5676 (1)	...
I(3)	0.4369 (1)	0.1998 (2)	0.4389 (1)	...
Ni	0.3708 (1)	0.2308 (2)	0.5031 (2)	...
P	0.3240 (2)	0.2365 (4)	0.3989 (4)	...
C(1)	0.4314 (7)	0.1839 (19)	0.8292 (16)	6.0 (7)
C(2)	0.4358 (7)	0.0964 (19)	0.8572 (16)	5.9 (7)
C(3)	0.4470 (8)	0.0293 (19)	0.8045 (18)	6.8 (8)
C(4)	0.4525 (8)	0.0512 (20)	0.7230 (18)	7.2 (8)
C(5)	0.4463 (7)	0.1339 (20)	0.6905 (16)	6.2 (7)
C(6)	0.4369 (7)	0.2075 (19)	0.7464 (18)	6.5 (7)
C(7)	0.4188 (8)	0.2366 (16)	1.0145 (15)	4.9 (7)
C(8)	0.4523 (8)	0.2714 (19)	1.0605 (18)	7.1 (8)
C(9)	0.4506 (9)	0.2388 (20)	1.1485 (18)	7.6 (8)
C(10)	0.4208 (9)	0.1770 (21)	1.1756 (17)	7.4 (8)
C(11)	0.3934 (8)	0.1388 (20)	1.1207 (20)	7.8 (8)
C(12)	0.3902 (8)	0.1723 (20)	1.0379 (17)	6.9 (8)
C(13)	0.3651 (6)	0.3140 (15)	0.8741 (14)	3.4 (6)
C(14)	0.3455 (8)	0.2884 (16)	0.8001 (16)	5.4 (7)
C(15)	0.3081 (8)	0.3255 (19)	0.7874 (15)	6.0 (7)
C(16)	0.2879 (7)	0.3821 (19)	0.8412 (17)	6.6 (7)
C(17)	0.3104 (8)	0.4089 (17)	0.9165 (16)	5.8 (7)
C(18)	0.3472 (7)	0.3715 (17)	0.9332 (15)	5.1 (6)
C(19)	0.4536 (8)	0.3745 (18)	0.8896 (14)	4.9 (6)
C(20)	0.4923 (9)	0.3521 (20)	0.8792 (17)	7.5 (8)
C(21)	0.5184 (9)	0.4188 (23)	0.8686 (18)	8.3 (9)
C(22)	0.5060 (9)	0.5088 (22)	0.8603 (17)	7.8 (8)
C(23)	0.4668 (10)	0.5296 (21)	0.8886 (18)	8.5 (9)
C(24)	0.4386 (9)	0.4570 (23)	0.8932 (18)	8.5 (9)
C(25)	0.3089 (7)	0.1255 (16)	0.3641 (13)	4.4 (6)
C(26)	0.2676 (7)	0.1009 (18)	0.3574 (15)	5.4 (7)
C(27)	0.2591 (7)	0.0101 (18)	0.3323 (14)	5.0 (6)
C(28)	0.2911 (7)	−0.0428 (15)	0.3077 (14)	4.1 (6)
C(29)	0.3299 (7)	−0.0205 (15)	0.3133 (13)	4.0 (6)
C(30)	0.3386 (7)	0.0672 (16)	0.3424 (14)	4.3 (6)
C(31)	0.2791 (6)	0.2930 (16)	0.4278 (14)	3.7 (6)
C(32)	0.2599 (7)	0.2697 (16)	0.4984 (15)	4.4 (6)
C(33)	0.2298 (8)	0.3202 (19)	0.5208 (15)	5.7 (7)
C(34)	0.2133 (7)	0.3863 (18)	0.4763 (16)	5.9 (7)
C(35)	0.2314 (7)	0.4095 (15)	0.3999 (14)	4.2 (6)
C(36)	0.2636 (7)	0.3618 (16)	0.3788 (13)	4.0 (6)
C(37)	0.3372 (7)	0.2940 (17)	0.3056 (15)	4.6 (6)
C(38)	0.3603 (8)	0.3646 (20)	0.3159 (17)	6.6 (7)
C(39)	0.3715 (9)	0.4218 (21)	0.2453 (22)	9.7 (10)
C(40)	0.3550 (9)	0.3945 (22)	0.1669 (19)	8.5 (9)
C(41)	0.3315 (7)	0.3158 (17)	0.1600 (14)	4.2 (6)
C(42)	0.3227 (9)	0.2683 (22)	0.2292 (19)	3.7 (9)

Anisotropic Thermal Parameters[c]

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
As	5.0 (2)	4.0 (2)	4.0 (2)	0.1 (2)	0.2 (1)	0.3 (1)
I(1)	8.4 (1)	6.3 (1)	5.7 (1)	−1.8 (1)	−1.1 (1)	2.1 (1)
I(2)	12.0 (2)	5.1 (1)	6.6 (1)	−0.8 (1)	−0.4 (1)	−1.8 (1)
I(3)	6.7 (1)	9.3 (2)	6.4 (1)	0.1 (1)	0.4 (1)	−1.3 (1)
Ni	5.8 (2)	4.0 (2)	3.6 (2)	−0.9 (2)	−0.4 (2)	0.1 (2)
P	4.7 (4)	3.1 (4)	3.8 (4)	−0.2 (3)	0.9 (3)	0.0 (3)

[a] The numbers in parentheses here and in succeeding tables are the estimated standard deviations of the least significant digit(s). [b] The atom labeling conforms to Figures 2 and 4. [c] The form of the anisotropic thermal ellipsoid is: $\exp[-0.25\cdot(B_{11}b_1{}^2h^2 + B_{22}b_2{}^2k^2 + B_{33}b_3{}^2l^2 + 2B_{12}b_1b_2hk + 2B_{13}b_1b_3hl + B_{23}b_2b_3kl)]$, where b_i is the ith reciprocal axis.

TABLE III

PHENYL RING BOND DISTANCES AND ANGLES

Bond Distances (Å)

C(1)–C(2)	1.395 (40)	C(22)–C(23)	1.410 (45)
C(1)–C(6)	1.412 (39)	C(23)–C(24)	1.453 (46)
C(3)–C(2)	1.382 (39)	C(25)–C(26)	1.451 (36)
C(3)–C(4)	1.386 (42)	C(25)–C(30)	1.388 (34)
C(5)–C(4)	1.362 (42)	C(27)–C(26)	1.447 (37)
C(5)–C(6)	1.471 (40)	C(29)–C(28)	1.416 (35)
C(7)–C(8)	1.436 (38)	C(29)–C(28)	1.359 (35)
C(7)–C(12)	1.430 (38)	C(29)–C(30)	1.425 (34)
C(9)–C(8)	1.514 (42)	C(31)–C(32)	1.384 (33)
C(9)–C(10)	1.454 (43)	C(31)–C(36)	1.395 (33)
C(11)–C(10)	1.391 (42)	C(33)–C(32)	1.333 (36)
C(11)–C(12)	1.437 (42)	C(33)–C(34)	1.338 (38)
C(13)–C(14)	1.407 (34)	C(35)–C(34)	1.449 (35)
C(13)–C(18)	1.441 (33)	C(35)–C(36)	1.360 (33)
C(15)–C(14)	1.397 (37)	C(37)–C(38)	1.326 (38)
C(15)–C(16)	1.415 (38)	C(37)–C(42)	1.373 (34)
C(17)–C(16)	1.475 (37)	C(39)–C(38)	1.494 (44)
C(17)–C(18)	1.389 (36)	C(39)–C(40)	1.433 (47)
C(19)–C(20)	1.375 (41)	C(41)–C(40)	1.427 (40)
C(19)–C(24)	1.339 (43)	C(41)–C(42)	1.374 (33)
C(21)–C(20)	1.350 (45)	C(21)–C(22)	1.414 (47)
	Av 1.407 (7)[a]		

Bond Angles (deg)

C(6)–C(1)–C(2)	122.0 (25)	C(21)–C(22)–C(23)	120.0 (28)
C(1)–C(2)–C(3)	120.8 (25)	C(22)–C(23)–C(24)	118.2 (28)
C(2)–C(3)–C(4)	118.0 (26)	C(23)–C(24)–C(19)	116.0 (27)
C(3)–C(4)–C(5)	124.3 (27)	C(30)–C(25)–C(26)	122.0 (22)
C(4)–C(5)–C(6)	118.5 (25)	C(25)–C(26)–C(27)	116.3 (22)
C(5)–C(6)–C(1)	116.2 (25)	C(26)–C(27)–C(28)	117.3 (22)
C(12)–C(7)–C(8)	129.8 (24)	C(27)–C(28)–C(29)	126.6 (22)
C(7)–C(8)–C(9)	108.5 (23)	C(28)–C(29)–C(30)	116.0 (21)
C(8)–C(9)–C(10)	122.9 (26)	C(29)–C(30)–C(25)	121.4 (22)
C(9)–C(10)–C(11)	121.7 (27)	C(36)–C(31)–C(32)	118.7 (21)
C(10)–C(11)–C(12)	118.9 (27)	C(31)–C(32)–C(33)	118.2 (23)
C(11)–C(12)–C(7)	117.1 (24)	C(32)–C(33)–C(34)	125.6 (25)
C(18)–C(13)–C(14)	122.4 (21)	C(33)–C(34)–C(35)	117.4 (23)
C(13)–C(14)–C(15)	114.7 (22)	C(34)–C(35)–C(36)	117.3 (21)
C(14)–C(15)–C(16)	127.4 (24)	C(35)–C(36)–C(31)	122.4 (21)
C(15)–C(16)–C(17)	115.3 (23)	C(42)–C(37)–C(38)	122.0 (24)
C(16)–C(17)–C(18)	119.2 (23)	C(37)–C(38)–C(39)	121.9 (26)
C(17)–C(18)–C(13)	120.7 (22)	C(38)–C(39)–C(40)	114.6 (27)
C(24)–C(19)–C(20)	126.7 (27)	C(39)–C(40)–C(41)	120.4 (27)
C(19)–C(20)–C(21)	117.9 (28)	C(40)–C(41)–C(42)	120.2 (23)
C(20)–C(21)–C(22)	120.5 (28)	C(41)–C(42)–C(37)	120.8 (22)
	Av 119.9 (6)[a]		

[a] The standard deviations of the averages were calculated from $\sigma = (\Sigma_i\Delta_i{}^2/m(m-1))^{1/2}$.

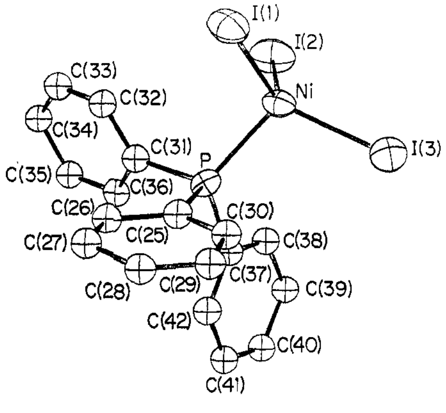

Figure 2.—The triiodo(triphenylphosphine)nickelate(II) anion.

Anion Geometry.—The important bond angles and distances internal to $Ni(C_6H_5)_3PI_3^-$ are set out in Table IV. The anion and the atom-labeling scheme are illustrated in Figure 2. The pseudo-tetrahedral structure expected on the basis of spectral and magnetic studies[14] is substantiated. The bond angles at the nickel atom are all quite near the tetrahedral angle (109.5°) with the I–Ni–I angles averaging slightly greater than this angle while the I–Ni–P angles average slightly less than this angle. This behavior is presumably due to the fact that I–I steric repulsion is greater than that for I–P. The stereoview of the anion viewed down the Ni–P bond axis (Figure 3) shows the anion to have nearly C_{3v} symmetry (excluding the phenyl rings). It is seen that the iodine atoms are rotationally staggered with respect to the phenyl groups. Furthermore, the phenyl groups are "pitched," presumably to maximize attractive van der Waals forces and to minimize steric repulsion.

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[14] F. A. Cotton, O. D. Faut, and D. M. L. Goodgame, *J. Am. Chem. Soc.*, **83**, 344 (1961).

TABLE IV

IMPORTANT ANION AND CATION BOND DISTANCES AND ANGLES

Anion Bond Distances (Å)			
Ni–I(1)	2.538 (4)	P–C(25)	1.825 (25)
Ni–I(2)	2.546 (4)	P–C(31)	1.822 (23)
Ni–I(3)	2.551 (4)	P–C(37)	1.814 (26)
Ni–P	2.284 (8)		
Anion Bond Angles (deg)			
I(1)–Ni–I(2)	109.9 (1)	Ni–P–C(25)	112.1 (8)
I(1)–Ni–I(3)	119.2 (2)	Ni–P–C(31)	113.2 (8)
I(2)–Ni–I(3)	112.8 (2)	Ni–P–C(37)	116.9 (8)
I(1)–Ni–P	102.1 (2)	C(25)–P–C(31)	106.0 (11)
I(2)–Ni–P	103.3 (2)	C(25)–P–C(37)	104.4 (11)
I(3)–Ni–P	107.6 (2)	C(31)–P–C(37)	103.3 (11)
Cation Bond Distances (Å)			
As–C(1)	1.885 (28)	As–C(13)	1.904 (21)
As–C(7)	1.941 (24)	As–C(19)	1.922 (27)
Cation Bond Angles (deg)			
C(1)–As–C(7)	111.2 (11)	C(19)–As–C(1)	109.3 (11)
C(7)–As–C(13)	107.3 (10)	C(1)–As–C(13)	109.1 (10)
C(13)–As–C(19)	110.4 (10)	C(7)–As–C(19)	109.6 (10)

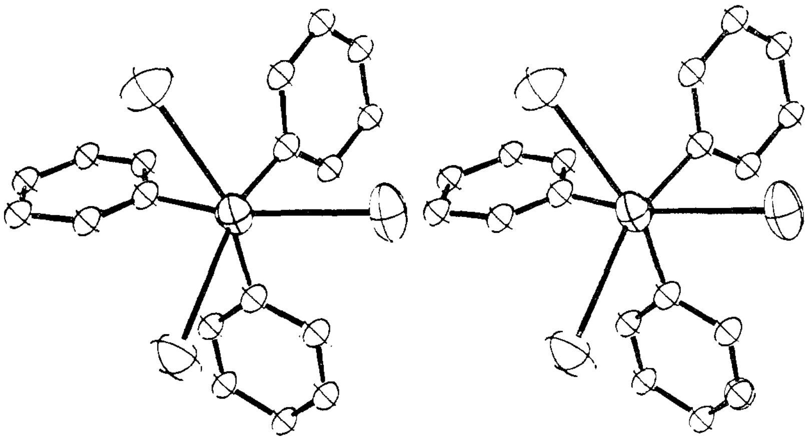

Figure 3.—Stereopair showing the anion viewed down the nickel-phosphorus bond ("C_3" axis).

This "pitch" can be described quantitatively by the angle made by the least-squares plane through the phenyl group with the plane of the nickel, phosphorus, and phenyl carbon atom to which the phosphorus atom is bonded. In terms of this definition the "pitches" are as follows: phenyl(25–30), 52.9°; phenyl(31–36), 50.6°; phenyl(37–42), 34.5°.

The Ni–P distance of 2.28 Å is in perfect agreement with that found[10] in another pseudo-tetrahedral nickel(II) complex, Ni[$(C_6H_5)_3P$]$_2$Cl$_2$. The Ni–I distances agree quite well among themselves and average 2.55 Å. As expected for tetrahedral coordination, this distance is less than that predicted (~2.67 Å) on the basis of covalent radii for a Ni–I distance in an octahedral nickel(II) complex. Provided one takes the tetrahedral covalent radii[15] for the halogens (Cl, 0.99 Å; Br, 1.11 Å; I, 1.28 Å), the present Ni–I distance is in excellent agreement with that predicted (2.56 Å) from the Ni–Cl distance (2.27 Å) in $NiCl_4^{2-}$ [9] and Ni[$(C_6H_5)_3P$]$_2$Cl$_2$,[10] when the difference in the Cl and I radii is taken into account. An exactly similar situation occurred[8] for Ni(quin)Br_3^-. Using these halogen radii and the observed Ni–X internuclear distances for the pseudo-tetrahedral nickel(II) species, a consistent tetrahedral covalent radius for nickel(II) of 1.27 Å is obtained, which may be compared with the octahedral radius of 1.39 Å.[16]

Cation Geometry.—The structure of the tetraphenylarsonium ion and the carbon atom labeling scheme are shown in Figure 4. A stereoview is provided by Figure 5. The bond distances and angles involving the arsenic atom are listed in Table IV. The phenyl groups are disposed in an almost perfect tetrahedral fashion about the arsenic atom. The six C–As–C angles average 109.5° and no deviation from the tetrahedral angle of more than 2.3° is observed. The average As–C bond length of 1.91 Å observed here is in excellent agreement with values found (1.90[17] and 1.91[18] Å) in two recent crystal structure determinations involving the tetraphenylarsonium ion. When the phenyl rings are considered, the cation possesses no over-all symmetry. The phenyl rings apparently are free to achieve that rotational orientation with respect to their As–C bond axes which produces the most favorable crystal-packing energy. This was found to be the case in two other

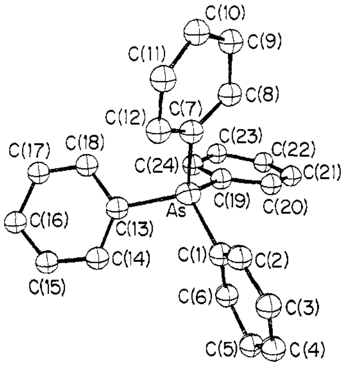

Figure 4.—The tetraphenylarsonium ion.

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(15) L. Pauling, "The Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960, p 246, Table 7-13. The use of the perhaps more appropriate single-bond covalent radii for the halogens produces less satisfactory predictions for the Ni–Br and Ni–I distances. It may be that the use of tetrahedral covalent radii in some way compensates for the increasing difference in nickel(II) and halogen electronegativities in the series Cl to I.

(16) See ref 15, p 249, Table 7-15.

(17) F. A. Cotton and S. J. Lippard, *Inorg. Chem.*, **5**, 416 (1966).

(18) T. E. Hopkins, A. Zalkin, D. H. Templeton, and M. G. Adamson, *ibid.*, **5**, 1427 (1966).

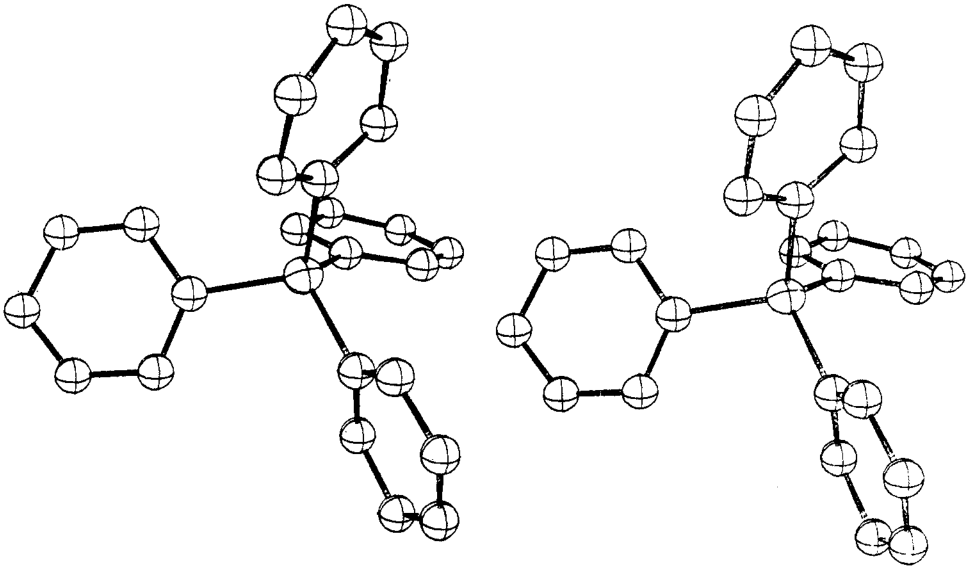

Figure 5.—Stereopair showing the tetraphenylarsonium ion.

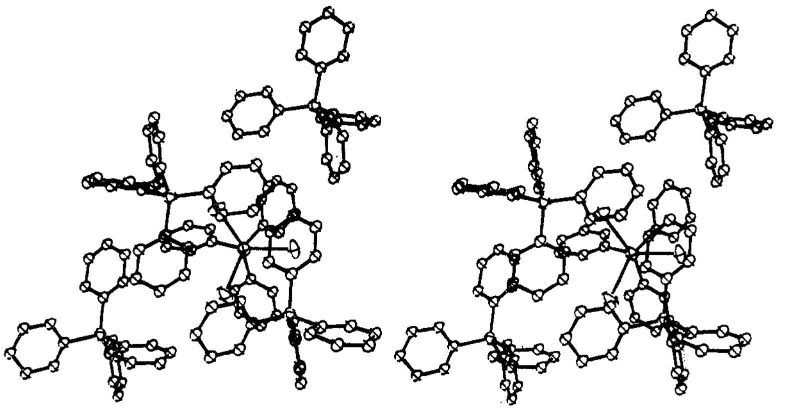

Figure 6.—Stereopair showing an anion and the four nearest neighbor cations as they are juxtaposed in the unit cell viewed down the "C_3" axis of the anion.

recently determined structures[17,18] despite the fact that the tetraphenylarsonium ion exhibited some crystallographically imposed symmetry in some earlier X-ray determinations.[19] The orientation of the phenyl rings in the present case is depicted in Figure 5. Quantitatively, the phenyl ring orientation may be described by the dihedral angles formed by least-squares planes through the various groups as was done elsewhere.[18] The phenyl groups are labeled as follows (according to the carbon atoms comprising them): I(1–6), II(7–12), III(13–18), IV(19–24). The dihedral angles between the various planes are: I–II, 67.1°; I–III, 63.2°, I–IV, 69.4°; II–III, 77.1°; II–IV, 80.9°; III–IV, 67.2°.

Over-all Structure.—In view of the previous estimate[6] of interionic distance in ion pairs of the cobalt analog of the present compound, the mode and proximity of approach of counterions in the solid state is of interest. An interionic distance estimate for ions of an ion pair of ~9.0 Å (Co–As distance) was made[6] on the basis of a model in which the cation approached the anion along the latter's "C_3" axis on the side opposite the bulky triphenylphosphine ligand. No other mode of counterion approach was considered. In the solid state, if one concentrates attention on a single anion at say, x, y, z (Table II), there are four nearest neighbor tetraphenylarsonium cations. These are symmetry related to one another by the following equivalent positions: A, x, y, z; B, $1 - x, y, 1^1/_2 - z$; C, $x, -y, z - {}^1/_2$; D, $x, 1 - y, z - {}^1/_2$. The stereoview provided by Figure 6 illustrates the juxtaposition of the four nearest neighbor cations with respect to an anion when viewed down the latter's "C_3" axis on the side away from the triphenylphosphine. It is immediately evident that no cation lies directly along this axis. Cations A and B, which are nearest the viewer, occupy positions about equally displaced on either side of the "C_3" axis. The Ni–As distances of 6.64 and 7.33 Å represent the closest anion–cation approach in the crystal. However, the two other nearest neighbor cations, C and D, approach to within 7.96 and 7.75 Å, respectively from the "side" of the anion, *i.e.*, between the iodine atoms and the triphenylphosphine. The next nearest anion–cation distance (not shown) is 9.98 Å.

Several features of the solid-state structure may have

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[19] (a) R. C. L. Mooney, *J. Am. Chem. Soc.*, **62**, 2955 (1940); (b) R. C. L. Mooney-Slater, *Acta Cryst.*, **12**, 187 (1959); (c) B. Zaslow and R. E. Rundle, *J. Phys. Chem.*, **61**, 490 (1957).

some bearing on the solution pmr studies. Close counterion approach to within 6.6–8.0 Å is achieved from four different directions and is to be compared with the 9.0 Å estimate made from solution studies. While none of the cations approaches exactly along the "C_3" axis of the anion, the two closest, A and B, do approach from the side opposite the triphenylphosphine. Cations C and D come almost as close *via* a sidewise approach. This latter mode of ion-pair contact was not considered in the analysis of the solution data. It is to be noted that close approach from the triphenylphosphine end of the anion does not occur.

While the factors which affect the structures of ion pairs in solution (counterion attraction and solvation considerations) and solid-state structure (Madelung energy and favorable packing) are by no means identical, the present results indicate that some caution must be exercised when interpreting solution-state pmr results in terms of quantitative interionic distance estimates. The assumption that the sole mode of counterion approach is along the "C_3" axis of the anion is particularly questionable. However, it is not unreasonable that this is perhaps the dominant situation in ion pairs in solution. A reevaluation of the solution-state data in terms of alternative modes of approach would not be fruitful in view of the additional unknown parameters involved.

Acknowledgments.—R. P. T. and W. D. H. wish to thank the Lawrence Radiation Laboratory for its hospitality during the course of this work. We are indebted to Mr. B. G. DeBoer, Dr. M. G. B. Drew, and Mrs. H. Ruben for considerable help and useful discussion involving all aspects of this work.

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The Crystal and Molecular Structure of Chlorotriphenylphosphonium Bis(*cis*-1,2-bis(trifluoromethyl)ethene-1,2-dithiolato)gold, $[PCl(C_6H_5)_3][Au(S_2C_2(CF_3)_2)_2]$

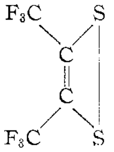
BY JOHN H. ENEMARK AND JAMES A. IBERS

Received July 1, 1968

An X-ray diffraction study has shown that the product of the reaction between *cis*-bis(trifluoromethyl)-1,2-dithietene, $S_2C_2(CF_3)_2$, and chlorotriphenylphosphinegold, $(C_6H_5)_3PAuCl$, is the unexpected compound chlorotriphenylphosphonium bis(*cis*-1,2-bis(trifluoromethyl)ethene-1,2-dithiolato)gold, $[PCl(C_6H_5)_3][Au(S_2C_2(CF_3)_2)_2]$. The compound crystallizes in space group C_{2h}^5-$P2_1/n$ of the monoclinic system with four molecules in a cell of dimensions $a = 13.166$ (10), $b = 11.623$ (9), $c = 20.486$ (15) Å, and $\beta = 91.21$ (2)°. The calculated and observed densities are 2.01 and 2.03 (2) g cm^{-3}. Least-squares refinement of the structure has led to a final value of the conventional R factor of 0.070 for the 1858 reflections, observed by counter methods, for which $F_o^2 > 3\sigma(F_o^2)$. The structure consists of well-separated ions. The coordination about the Au atom of the anion is square planar, with the four independent Au–S distances ranging from 2.282 (8) to 2.296 (8) Å. The S–Au–S angles range from 89.6 (3) to 90.6 (3)°. The S···S intra- and interligand distances, which range from 3.225 (11) to 3.254 (11) Å, are considerably longer than those previously observed in square-planar dithiolate complexes. The chlorotriphenylphosphonium cation has nearly tetrahedral geometry; the P–Cl distance is 1.98 (1) Å and the three P–C distances are in the range 1.76–1.77 (2) Å.

Introduction

Because of a general interest in five-coordinate transition metal compounds and in *cis*-1,2-disubstituted ethene-1,2-dithiolato complexes (hereafter called dithiolate complexes), we were intrigued by the report of a novel five-coordinate gold complex $[(C_6H_5)_3P\cdot Au(S_2C_2(CF_3)_2)_2]Cl$. This compound was prepared by Davison, Howe, and Shawl[1] from the reaction of bis(trifluoromethyl)-1,2-dithietene (I) with chlorotriphenylphosphinegold, $(C_6H_5)_3PAuCl$. The results, reported here, of an X-ray diffraction study of this compound indicate that it does not contain five-coordinate gold but that an unusual reaction has occurred to yield the chlorotriphenylphosphonium cation and the square-planar bis(*cis*-1,2-bis(trifluoromethyl)ethene-1,2-dithiolato)gold anion.[2]



I

Collection and Reduction of the X-Ray Data

Pale green crystals of what proved to be $[PCl(C_6H_5)_3][Au(S_2C_2(CF_3)_2)_2]$ were kindly supplied by Professor A. Davison and were used without further recrystallization. Preliminary precession photographs taken with Cu Kα radiation indicated that the crystals

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(1) A. Davison, D. V. Howe, and E. T. Shawl, *Inorg. Chem.*, **6**, 458 (1967).

(2) This nomenclature follows that previously used (R. Eisenberg and J. A. Ibers, *ibid.*, **5**, 411 (1966), particularly footnote 20), and it is not meant to imply a particular valence bond description of the complex.