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The Crystal and Molecular Structures of Some Condensation Products of Succinaldehyde and *p*-Bromophenylhydrazine

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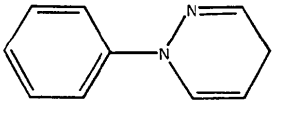
The crystal structures of two isomeric condensation products ($C_{20}H_{18}Br_2N_4$) of succinaldehyde and *p*-bromophenylhydrazine have been determined using three-dimensional diffractometer-collected X-ray data. Both compounds crystallize in the space group $P2_1/c$, with 4 molecules in the unit cell. The cell dimensions are $a=12{\cdot}20$ (3), $b=10{\cdot}12$ (1), $c=16{\cdot}37$ (2) Å, $\beta=110{\cdot}4$ (1)°, and $a=7{\cdot}429$ (3), $b=15{\cdot}444$ (8), $c=15{\cdot}991$ (9) Å, $\beta=93{\cdot}47$ (6)°. The compounds were found to be the diastereomeric racemates (4a*RS*, 4b*SR*,13b*RS*)-12-bromo-1-(*p*-bromophenyl)-1,4a,4b,5,6,13b-hexahydro-4*H*-dipyridazino[1,6-*a*:4,3-*c*]-quinoline and (4a*RS*,4b*RS*,13b*RS*)-12-bromo-1-(*p*-bromophenyl)-1,4a,4b,5,6,13b-hexahydro-4*H*-dipy-ridazino-[1,6-*a*:4,3-*c*]quinoline. The crystal structure of one of the enantiomers of the former, which spontaneously resolved from the solution of the racemate, was also determined. The enantiomer crystallizes in the space group $P2_12_12_1$ with $Z=4$ and cell parameters $a=13{\cdot}11$ (3), $b=14{\cdot}96$ (5), $c=9{\cdot}41$ (1) Å. Only small differences in the conformations of the molecules were found, but the packing of the molecules are quite different.

Introduction

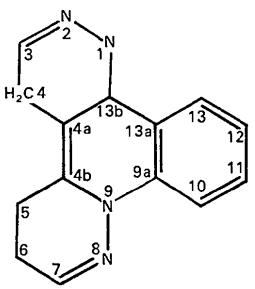
Ciamician & Zanetti (1890) determined a condensation product of succinaldehyde and phenylhydrazine to have the molecular formula $C_{20}H_{20}N_4$, and the constitutional formula was proposed to be 'a double molecule of a pyridazine derivative'. Desaty, Hadžija & Keglević (1965) proposed the structure of the condensation product on the basis of spectral and chemical evidence to be the dihydropyridazine derivate $C_{10}H_{10}N_2$.

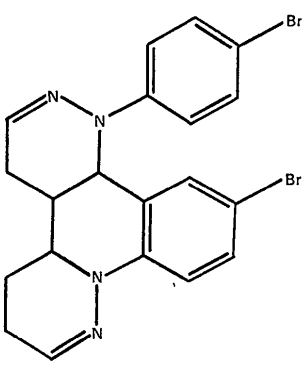


However, recent investigations (Hjeds & Larsen, 1971) showed that the condensation product consisted

of two compounds. The mass spectra of both compounds suggested an ion of $m/e=316$, leading to the formula $C_{20}H_{20}N_4$. The compounds were also investigated by ^1H nuclear magnetic resonance spectroscopy, but as no conclusive evidence of the structures was obtained, X-ray analyses of the *p*-bromo derivatives of both of them were performed.

The compounds, hereinafter referred to as HJBR-1 and HJBR-2, were found to be diastereomeric racemates of a derivative of the new ring system 4*H*-dihydropyridazine[1,6-*a*:4,3-*c*]quinoline.





The names of HJBR-1 and HJBR-2, respectively, are: (4a*RS*,4b*SR*,13b*RS*)-12-bromo-1-(*p*-bromophenyl)-1,4a,4b,5,6,13b-hexahydro-4*H*-dipyridazino-[1,6-*a*:4,3-*c*] quinoline and (4a*RS*,4b*RS*,13b*RS*)-12 bromo-1-(*p*-bromophenyl)-1,4a,4b,5,6,13b-hexahydro-4*H*-dipyridazino-[1,6-*a*:4,3-*c*]quinoline.

A possible mechanism for the ring closure is given in the preliminary report on the results of the structure analyses (Hjeds & Larsen, 1971).

Structure determination of one of the spontaneously resolved enantiomers of HJBR-1 was also performed.

Experimental

The condensation products HJBR-1 and HJBR-2 ($C_{20}H_{18}Br_2N_4$) crystallize from a mixture of benzene and ethanol 3:1 as monoclinic needles, space group $P2_1/c$. In addition orthorhombic crystals were sometimes obtained from the solution of HJBR-1 as well as from the solution of HJBR-2. This phenomenon was originally ascribed to polymorphism, but later, when HJBR-1 and HJBR-2 were determined to be diastereomeric racemates, it was evident that spontaneous resolution had occurred on crystallization from the racemic solutions. A similar phenomenon was recently reported by Cheng, Koo, Mellor, Nyburg & Young (1970).

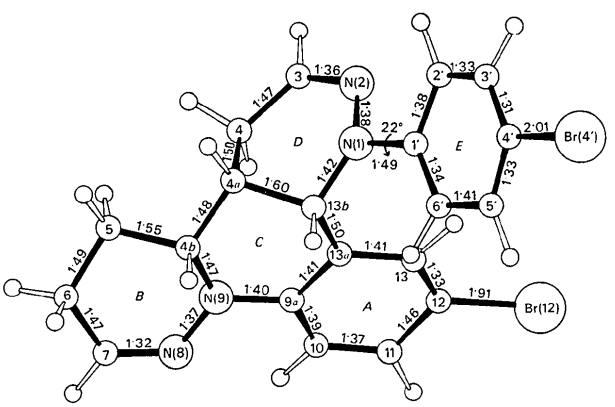


Fig. 1. The molecular structure of HJBR-1.

Table 1. *Crystal data for* HJBR-1, HJBR-1*a*, HJBR-2 *and* HJBR-2*a*

	HJBR-1	HJBR-1*a*	HJBR-2	HJBR-2*a*
Mol. Formula	$C_{20}H_{18}Br_2N_4$	$C_{20}H_{18}Br_2N_4$	$C_{20}H_{18}Br_2N_4$	$C_{20}H_{18}Br_2N_4$
M.W.	474·2	474·2	474·2	474·2
m.p.	264–66° (decomp.)	263–64° (decomp.)	257–59° (decomp.)	248–51° (decomp.)
Space group	$P2_1/c$	$P2_12_12_1$	$P2_1/c$	$P2_12_12_1$
a (Å)	12·20 (3)	13·11 (3)	7·429 (3)	13·51
b (Å)	10·12 (1)	14·96 (5)	15·444 (8)	14·33
c (Å)	16·37 (2)	9·412 (10)	15·991 (9)	9·660
β (°)	110·4 (1)	90	93·47 (6)	90
V (Å^3)	1894	1845	1833	1870
Z	4	4	4	4
D_x g.cm^{-3}	1·66	1·71	1·72	1·68
Dm g.cm^{-3}	1·66	1·70	1·70	1·69
μ(Mo $K\alpha$) cm^{-1}	45·5	46·7	47·0	46·1
Crystal size (mm)	0·2 × 0·3 × 1·0	0·2 × 0·2 × 1·0	0·24 × 0·34 × 0·70	
Rotation axis (=needle axis)	*b*	*b*	*a*	*b*

Table 2. *Observed and calculated structure factors for* HJBR-1

The columns are h, 10 $|F_o|$ and 10F_c.

H,-3,1
3 448 -397
-1 237 279
-2 155 -275
-3 240 237

H,-3,3
0 291 -330
-4 306 307

H,-3,4
-3 154 199

H,-3,5
0 263 249
-3 180 164

H,-3,9
-2 193 -171

H,-3,8
-1 166 190
-2 187 -142
-4 250 -257

H,-3,7
-4 192 192

H,-3,6
3 145 160
2 258 -279
1 188 188
-3 205 -189
-4 351 357

H,-3,5
-6 184 156
-3 154 178
-2 326 -333
-1 362 377
0 332 -342
3 261 -267

H,-3,4
4 208 -233
2 143 -140
0 171 -105
-1 134 173
-4 202 200

H,-3,3
-5 176 -143
-4 388 443
-3 383 -381
-2 214 229
-1 228 -193

H,-3,2
5 265 197
4 229 -173
3 219 274
1 384 -390
0 176 159
-1 434 -440
-2 217 240
-3 297 -303
-5 332 373

H,-3,1
-1 279 257
0 351 -360
1 601 611
2 373 -364
3 211 163
5 197 -217

H,-3,0
5 208 -152
1 278 235
0 156 131
-1 269 235

H,-7,0
-7 319 -311
-6 266 232
-5 626 -605
-2 269 -248
-1 214 -178
1 220 178
2 252 248
5 627 605
6 250 -242
7 332 311

H,-7,1
6 210 -221
5 330 -332
4 245 -243
3 330 299
2 146 -133
1 226 166
0 165 176
-1 270 240
-2 183 -133
-5 164 -150
-7 252 -266

H,-7,2
-2 202 222
-6 422 -407
-5 456 511
-4 693 -701
-3 536 527
-2 351 -372
0 342 402
1 639 -651
2 648 655
3 516 -508
6 241 -291
7 267 288

H,-7,3
6 269 259
4 208 211
3 154 -143
1 210 -210
-1 214 -235
-3 171 177
-4 153 -149
-5 146 216
-6 189 -265
-7 156 216

H,-7,4
-6 240 265
-2 296 251
2 226 -229
3 444 488
4 569 -562

H,-7,5
5 162 -95
1 282 322
-1 199 167

H,-7,6
-7 356 402
-6 349 -382
-5 619 640
-2 246 215
-1 483 -501
0 330 332
1 248 -262

H,-7,7
3 238 -227
-1 148 -128
-2 192 230
-5 185 -184
-6 223 -252

H,-7,8
-6 157 -114
-5 143 -99
-4 165 132
-3 308 -278
-2 165 119

H,-7,9
-1 162 118
-5 184 214
-6 164 150

H,-7,10
-5 172 -181
-3 215 -218
-2 168 126

H,-7,11
-3 185 -173

H,-6,12
-3 349 -308
-5 200 -154

H,-6,11
-4 267 -240
0 151 128

H,-6,10
2 149 -148
0 148 142
-3 372 341
-5 605 580
-6 359 -336

H,-6,9
-8 187 211
-4 325 -321
-3 267 201
-2 183 -133
2 247 267

H,-6,8
4 384 -360
3 240 194
2 207 -204
0 195 197
-1 295 -290
-2 303 306
-3 226 -196
-4 270 247
-5 156 -134

H,-6,7
-7 142 -131
-6 183 125
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0 165 -121
2 174 165
3 315 -313

H,-6,6
3 332 -318
2 217 222
1 362 -368
-2 562 -529
-3 804 752
-4 520 -530
-5 394 406
-7 327 -314
-8 253 293
-9 175 -176

H,-6,5
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-6 243 280
-5 272 -277
-4 307 236
-3 143 130
-2 326 -294
-1 389 371
0 146 -199
1 130 170
2 133 -146
3 256 -291
4 248 225
5 418 -399

H,-6,4
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6 232 -197
5 360 344
4 399 -383
2 425 404
0 339 319
-1 265 -253
-2 324 332
-3 170 -157
-5 288 -324
-8 196 -176
-9 176 213

H,-6,3
-9 150 122
-8 149 -216
-7 210 147
-5 342 -365
-4 293 345
-3 266 -307
-1 234 255
0 180 -230
1 455 475
2 142 -94
3 242 280
5 329 -297
7 209 -219

H,-6,2
6 261 243
5 155 -155
4 526 496
3 357 -316
1 350 332
0 689 -759
-1 846 832
-2 927 -954
-3 301 348
-4 141 168
-5 480 -488
-6 208 266
-7 369 -331
-8 182 240

H,-6,1
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-4 298 324
-3 352 -345
-2 468 461
4 158 -159
6 224 202

H,-6,0
6 395 -426
7 421 409
5 233 -217
3 392 -373
2 459 419
1 126 -102
0 544 550

H,-5,0
1 265 -246
2 570 544
3 456 -429
4 1053 977
5 540 -506
6 273 266
7 173 -72
8 150 -196
9 187 225

H,-5,1
9 415 -413
8 406 402
7 177 -60
5 523 477
4 947 -871
3 1290 1186
2 395 -380
1 137 -183
0 530 577
-1 700 -700
-2 661 698
-3 743 -728
-4 190 154
-5 407 418
-6 347 -362

H,-5,2
-8 159 165
-6 270 239
-5 457 -464
-4 276 276
-3 431 -446
-2 221 193
-1 226 201
0 241 224
1 461 474
2 362 -343
3 316 296
4 433 -390
5 147 162
6 438 -399

H,-5,3
6 772 742
4 308 -271
3 221 230
2 205 -210
1 474 -487
0 251 -233
-1 820 -795
-2 366 398
-3 117 88
-4 419 -424
-5 328 347
-6 576 -603
-7 325 372
-9 174 -161

H,-5,4
-8 299 -257
-7 578 614
-6 393 -409
-5 621 624
-4 457 -422
0 453 460
1 521 -609
2 619 626
3 321 -351
4 258 253
7 227 241
8 215 -239

H,-5,5
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5 481 -440
3 446 399
2 423 -431
1 1003 1046
0 224 -211
-1 125 116
-2 463 444
-3 779 -740
-4 572 582
-5 273 -315
-8 165 -194
-9 160 186

H,-5,6
-8 203 194
-7 159 -141
-6 279 286
-5 358 -325
-4 164 82
-3 224 -175
0 181 -195
1 408 403
2 259 -287
3 255 238
5 274 248

H,-5,7
5 165 -168
4 267 237
3 162 -228
2 236 -258
-1 180 118
-2 120 126
-3 200 -115
-4 430 454
-5 157 93
-6 161 -158
-7 255 286
-8 178 -284
-9 227 261

H,-5,8
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-8 371 -358
-7 205 165
-6 301 -257
-3 331 -317
-2 229 267
0 270 279
1 289 -297
4 192 -177

H,-5,9
3 390 -328
1 160 157
0 391 -385
-1 340 292
-2 302 -284
-3 374 346
-4 579 540
-5 389 -356
-6 525 473
-7 319 -283

H,-5,10
-8 211 180
-6 164 127
-3 209 184
-2 176 -124
1 205 193
3 142 -69

H,-5,11
2 157 170
-2 247 -261
-4 160 -171
-5 197 -176
-8 246 215

H,-5,12
-4 154 195

H,-5,13
-1 196 130
-4 289 -263
-5 147 168

H,-4,14
-4 258 204
-6 170 80

H,-4,13
-5 222 -165
-4 320 309
-2 151 41

H,-4,12
1 209 170
-3 611 -579
-4 328 -371
-6 237 -225
-7 335 287

H,-4,11
-8 171 -137
-7 251 290
-6 259 -264
-5 149 129
-3 367 386
3 233 -352
4 267 204
2 405 -426
1 211 -191
-2 293 264
-3 204 -162
-4 231 294

H,-4,9
-9 302 296
-6 338 -311
-4 432 399
-3 562 -469
-2 211 209
1 206 211
2 379 -417
3 177 198

H,-4,8
2 157 147
1 389 -408
0 483 490
-1 328 -338
-2 118 -142
-3 225 328
-4 643 -562
-5 519 466
-6 303 -347
-7 363 408
-9 184 -266
-10 176 277

H,-4,7
-9 323 -336
-7 184 170
-6 337 -356
-5 437 418
-4 441 -424
-3 247 216
0 449 -411
1 179 -146
4 142 185
5 295 -330
6 221 290

H,-4,6
6 602 -536
5 399 353
4 245 -180
2 394 400
1 242 -235
0 243 194
-1 353 -372
-4 359 -315
-6 139 129
-7 267 -275
-9 137 -151

H,-4,5
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-3 552 -536
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0 501 543
2 177 93
3 675 642
4 679 -654
5 464 434
8 166 152

H,-4,4
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7 196 192
5 158 -117
4 493 440
3 320 -303
2 1138 1134
1 200 193
-1 734 676
-2 913 -858
-3 799 849
-4 792 -865
-6 245 255
-7 202 -382
-8 418 455
-9 249 -300

H,-4,3
-10 322 325
-9 484 -445
-8 675 648
-7 502 -505
-6 273 300
-5 231 255
-4 971 1015
-3 762 751
-2 804 -737
-1 362 347
0 319 -314
1 139 142
2 420 403
3 171 198
5 184 -222
6 358 346
7 242 -205
8 201 149
9 203 -164

H,-4,2
9 228 267
8 393 -382
7 417 372
4 559 516
3 296 -301
2 285 301
1 634 -629
0 553 -573
-1 251 252
-2 335 -348
-3 280 269
-4 552 585
-5 605 -594
-6 297 341
-7 179 -163
-9 250 281
-10 212 -236

H,-4,1
-10 226 -228
-9 289 265
-8 204 -174
-7 293 270
-4 299 260
-1 514 -484
0 810 833
1 743 -740
2 396 378
3 308 -288
5 649 612
6 818 -800
7 380 376

H,-4,0
10 228 -117
7 287 -266
6 370 320
5 680 -645
4 424 403
3 176 -194
2 384 -396
1 772 750
0 1585 1456

H,-3,0
2 1260 1145
3 1217 1105
4 570 -511
5 459 -450
6 294 230
7 415 -412
8 284 265
10 200 -153

H,-3,1
10 153 -164
5 388 -318
4 687 612
3 449 -421
2 1194 1113
1 1301 1195
0 701 743
-1 240 234
-2 1141 1152
-3 647 636
-4 855 -845
-5 1239 1119
-6 648 -629
-7 416 390
-9 229 -247
-10 378 370

H,-3,2
-9 293 253
-8 503 -482
-7 384 349
-5 378 -369
-4 740 781
-3 177 -196
-2 573 552
-1 319 312
0 373 -385
1 1078 1039
2 809 -793
4 363 343
5 755 -645
6 493 458
7 396 -334

Table 2 (*cont.*)

H,-3,2
9 290 263
H,-3,3
9 377 -360
8 223 236
7 150 -67
6 186 195
5 655 618
4 761 -720
3 848 847
2 1201 1124
1 525 556
0 551 -585
-1 415 -407
-2 709 696
-3 951 1057
-4 754 838
-5 553 -577
-6 331 374
-7 131 -135
-8 202 -168
H,-3,4
-10 266 -257
-9 154 178
-8 237 -214
-5 562 -531
-4 707 706
-3 155 -183
-1 373 401
0 1093 1130
2 341 -319
3 329 -322
4 614 537
5 343 -333
6 211 200
7 371 348
9 199 153
H,-3,5
8 160 -234
7 383 -399
6 176 -177
5 150 -159
3 161 -149
2 727 720
1 687 633
0 948 971
-1 778 -750
-2 245 283
-5 585 648
-6 319 -415
-7 456 597
-8 328 -458
-9 230 330
-10 170 -164
-11 210 -162
H,-3,6
-11 311 307
-10 283 -265
-9 160 186
-8 162 122
-7 263 -253
-6 160 273
-5 894 -831
-3 123 126
-2 587 -594
-1 629 604
0 384 -368
1 246 272
2 598 679
3 362 -336
4 282 257
5 268 -236
H,-3,7
6 397 366
3 313 314
2 125 -135
1 722 745
0 857 -861
-1 601 611
-2 416 -392
-3 300 -261
-4 285 278
-5 767 -684
-6 680 728
-7 313 -414
-8 234 285
H,-3,8
-7 339 -317
-6 390 366
-4 363 -322
-3 628 578
-2 220 -163
-1 281 276
1 162 -173
2 459 578
3 133 -87
6 163 -240
H,-3,9
2 191 122
1 300 -186
0 124 110
-1 348 -362
-2 131 96
-6 146 -151
-7 251 194
-8 317 -362
H,-3,10
-9 336 -256
-8 329 255
-7 393 -269
-5 200 189
-3 523 497
-2 146 -125
-1 179 -140
1 202 -206
2 149 110
H,-3,11
-1 308 294
-2 578 -566
-4 339 -278
-7 356 -266
-8 576 525
-10 190 251
H,-3,12
-8 177 128
-7 146 117
-6 240 -192
-5 236 242
-4 292 -253
-3 137 -140
-1 257 -255
0 215 138
1 160 -237
H,-3,13
-4 171 154
H,-3,15
-6 159 180
-2 309 -272
H,-2,14
-2 152 174
-3 266 301
-7 149 -150
H,-2,13
-9 250 280
-2 466 -478
H,-2,12
2 163 128
0 177 173
-2 145 -110
-3 173 -215
-4 290 -291
H,-2,11
-7 148 -79
-6 543 516
-5 457 -393
-4 672 585
-3 280 -240
-1 203 156
0 437 -485
1 309 429
2 235 -230
H,-2,10
-4 309 270
-5 306 -267
-8 157 -100
-10 167 -130
H,-2,9
-11 217 -341
-8 417 -390
-7 419 420
-6 378 -364
-5 365 355
-3 259 -264
0 141 -188
1 278 366
4 152 132
5 156 -254
H,-2,8
4 316 214
3 216 -220
-1 206 269
-2 214 -222
-3 405 399
-4 257 -267
-5 128 -151
-9 158 -288
H,-2,7
-10 324 -280
-9 527 -515
-7 414 379
-6 125 -124
-5 216 -194
-4 944 740
-3 682 -659
-2 973 953
-1 932 -930
0 377 422
1 151 315
2 335 -506
4 411 -573
5 159 312
H,-2,6
6 151 -204
3 190 142
2 123 -172
1 263 312
-1 180 -191
-2 672 617
-3 654 -570
-4 461 381
-5 252 -252
-6 126 -162
-7 143 212
-8 224 -247
-9 301 315
H,-2,5
-11 284 -306
-10 666 636
-9 632 -674
-8 432 446
-7 136 158
-6 692 -639
-5 1064 987
-4 1034 -962
-3 625 633
-2 125 83
-1 185 145
0 457 478
3 115 114
5 162 159
7 326 -354
8 282 306
H,-2,4
8 155 175
7 284 -320
6 330 289
5 209 -238
4 163 180
2 323 -321
1 366 368
0 268 -277
-1 557 559
-2 433 -366
-5 270 341
-8 258 229
-9 260 -232
-11 369 -353
H,-2,3
-11 295 310
-8 153 -145
-7 191 207
-6 191 -196
-5 554 537
-4 666 -593
-3 218 -229
-2 1400 1255
-1 1187 1146
0 1006 1046
1 2235 2351
3 252 260
4 794 -808
5 973 915
6 875 -849
7 699 653
9 284 -247
H,-2,2
7 330 333
5 159 184
4 386 -361
3 495 476
2 275 -284
1 2219 2139
0 256 -215
-1 1238 1067
-2 955 981
-3 263 -278
-5 1238 1218
-7 356 362
-8 304 -262
H,-2,1
-9 804 -710
-8 866 853
-7 851 -792
-6 689 650
-5 171 119
-4 700 -659
-3 1908 1721
-2 2320 2168
-1 718 666
0 541 -532
1 1705 1576
2 496 -436
3 481 -480
4 557 517
5 163 118
7 152 123
8 223 217
10 238 221
H,-2,0
9 208 -230
8 442 439
7 500 -461
5 580 -526
4 153 -125
3 158 161
2 768 -661
1 1480 1259
0 342 275
H,-1,0
2 880 -782
3 1190 1091
4 1154 1113
5 694 640
7 223 -239
8 285 280
9 258 -267
H,-1,1
9 245 193
8 316 -315
6 475 -449
5 694 -634
4 163 133
2 623 -590
-2 235 -191
-3 676 -567
-4 1255 1109
-5 1109 1001
-6 849 786
-7 253 -226
-9 362 332
-10 369 -373
-11 198 226
H,-1,2
-11 159 -120
-10 244 206
-9 168 146
-8 563 -496
-7 856 762
-6 509 -530
-5 251 261
-4 380 -355
-3 198 140
-2 664 596
1 203 118
2 456 440
3 324 -358
4 691 683
5 549 -521
6 705 674
8 191 -149
9 291 284
10 260 -235
H,-1,3
9 176 176
8 300 -271
7 542 534
4 806 735
2 1104 1071
1 1260 1328
0 471 -460
-1 1471 1311
-2 621 -613
-3 577 786
-4 341 -478
-5 166 257
-6 149 215
-7 299 -303
-8 160 150
H,-1,4
-11 373 349
-10 244 -227
-9 222 219
-8 146 147
-7 760 -752
-6 1205 1148
-4 1075 1020
-3 728 -621
-2 176 -224
-1 493 535
0 1939 2244
1 258 418
2 1135 1176
3 432 529
4 161 152
5 282 -262
6 499 507
8 285 296
H,-1,5
6 330 -278
5 340 339
4 250 -200
3 226 -243
2 108 110
1 655 -685
0 375 -375
-1 557 521
-2 600 -539
-3 753 707
-6 436 615
-7 455 -673
-8 337 437
-9 322 -357
H,-1,6
-10 181 -117
-9 410 425
-8 132 -176
-7 245 274
-6 218 -258
-5 264 283
-3 120 55
-2 531 -506
-1 160 131
3 383 -538
4 272 357
5 146 -137
6 209 -237
7 190 252
8 212 -339
H,-1,7
7 222 225
6 318 -279
5 329 299
1 845 -917
0 737 777
-1 565 -619
-2 301 310
-4 1711 1443
-6 405 -415
-7 131 160
-10 318 378
H,-1,8
-11 186 -107
-9 510 -440
-8 610 642
-7 645 -630
-6 689 638
-3 907 880
-2 569 -570
-1 418 426
0 309 -408
1 131 244
H,-1,9
4 205 -111
3 284 292
2 122 -95
1 121 159
0 225 236
-1 174 -171
-2 151 138
-3 501 465
-4 217 187
-5 297 274
-6 151 -180
-7 130 96
-8 205 215
-9 490 -601
H,-1,10
-10 415 -394
-8 151 -176
-7 232 234
-6 284 -262
-5 213 168
-3 223 203
-2 250 233
-1 468 -513
0 230 341
1 305 -434
H,-1,11
3 198 239
2 203 -119
7 167 -111
6 198 143
5 536 523
4 828 -869
3 353 395
2 854 1021
1 335 346
0 290 334
-1 1934 1879
-2 2242 2038
-3 1996 1971
-4 630 665
-5 101 -162
-6 672 -796
-7 861 991
-8 803 -860
-9 419 395
-10 228 -182
H,-1,12
-10 354 318
-9 265 -279
-8 307 302
-5 162 126
-4 322 -325
-2 230 -268
H,-1,13
1 222 139
0 157 -174
-7 151 124
H,-1,14
-7 192 205
-3 143 -117
-2 171 153
-1 261 -307
0 148 200
H,0,14
1 220 -208
-1 215 -235
-2 267 316
-3 350 313
-4 137 -86
-5 352 308
-6 168 -80
H,0,12
2 231 219
-1 265 277
-2 386 -376
-3 248 207
-4 134 146
-6 371 338
-7 419 -390
-8 366 335
-10 146 -119
H,0,10
5 227 -195
4 346 334
3 337 -315
2 446 441
1 178 -173
0 303 -342
-1 233 227
-2 620 -626
-3 788 754
-4 131 118
-7 157 -105
-8 164 97
-9 210 -160
H,0,8
2 334 -347
1 534 592
0 593 -577
-1 282 279
-2 428 427
-3 919 -901
-4 1064 1007
-5 899 -780
-6 594 576
-7 180 -258
-8 435 -481
-9 380 499
-10 383 -488
-11 154 284
H,0,6
7 326 -326
6 572 621
5 614 -698
4 534 570
2 296 -363
1 405 447
0 630 -782
-1 895 949
-2 470 -421
-4 577 -483
-5 171 -187
-9 253 363
-10 670 763
H,0,4
7 167 -111
6 198 143
5 536 523
4 828 -869
3 353 395
2 854 1021
1 335 346
0 290 334
-1 1934 1879
-2 2242 2038
-3 1996 1971
-4 630 665
-5 101 -162
-6 672 -796
-7 861 991
-8 803 -860
-9 419 395
-10 228 -182
H,0,2
10 285 300
9 538 -574
8 717 675
7 735 -743
6 905 893
4 456 -381
3 876 831
2 1273 1146
-3 109 -200
-4 144 -137
-5 822 811
-7 360 -289
-8 290 254
-9 684 -691
-10 487 432
-11 398 -408
H,0,0
8 270 -205
7 266 252
6 2095 2003
5 750 781
4 1067 -989
H,-11,2
-4 202 144
1 217 148
2 188 -134
H,-11,3
-2 166 -96
H,-11,6
-1 203 207
H,-10,10
-5 221 -138
H,-10,9
-2 180 114
H,-10,6
4 170 79
-3 192 -205
-5 157 -115
H,-10,5
-8 183 -159
-6 167 -108
-3 210 -170
-2 196 192
0 173 160
4 169 -142
5 240 224
H,-10,4
-2 188 118
H,-10,3
-3 160 211
-2 155 -86
-1 159 162
H,-10,2
3 161 178
-1 254 -300

Table 2 (*cont.*)

H,-10,2	-6 194 -175	10 214 173	-10 189 -188	-7 243 -220	H,-3,3	H,-1,2
-2 164 158	-7 228 182	11 232 -227		-9 206 -150	11 241 -263	-13 213 -239
	-8 160 -135		H,-6,4		10 232 224	-12 302 279
H,-10,1		H,-7,1	-10 338 -403	H,-5,14		
-1 180 -183	H,-9,11	8 174 76	-11 178 273	-9 154 -126	H,-3,5	H,-1,4
0 187 224	-9 202 -102	-8 181 264		-7 174 -105	-12 196 181	-12 200 -220
2 176 159			H,-6,3	-3 205 118	-13 199 -250	
3 163 -124	H,-8,10	H,-7,2	-10 170 -186	-1 191 102		H,-1,5
6 216 -184	-4 217 218	-10 194 212			H,-3,7	11 165 -139
		-9 282 -302	H,-6,1	H,-4,17	-12 163 -194	10 251 231
H,-10,0	H,-8,9		-10 183 -129	-7 156 -126		-12 197 -207
6 165 -172	-9 201 -116	H,-7,3	-9 285 245		H,-3,8	-13 239 212
4 180 -133	-5 179 176	9 162 119	11 186 138	H,-4,15	-12 193 -110	-14 190 -156
3 152 141	-4 196 -151			-4 179 -154		
0 330 393	-3 166 191	H,-7,4	H,-6,0	2 163 147	H,-3,12	H,-1,6
-3 158 141	2 173 91	9 217 -233	10 221 -180		-10 187 190	-14 164 145
-6 189 -172			9 256 338	H,-4,12		
-7 159 -112	H,-8,8	H,-7,6	-9 330 338	-9 227 210	H,-3,15	H,-1,8
	-5 154 -168	-11 254 -287			-6 176 -146	-13 250 199
H,-9,0		-8 196 -182	H,-5,0	H,-4,11	-9 175 -142	-12 191 -215
6 162 -134	H,-8,7	5 200 169	-10 279 255	-12 194 195	-10 156 103	
	-9 194 163		10 318 -255	-11 202 -197		H,-1,9
H,-9,1	-6 300 349	H,-7,7			H,-3,16	-12 160 108
8 186 -142	5 180 132	-9 154 210	H,-5,1	H,-4,8	-3 186 -171	
5 395 -428	6 167 -178		10 267 290	-11 207 -183		H,-1,12
4 188 247		H,-7,8			H,-2,15	-11 208 -173
	H,-9,6	4 154 -67	H,-5,2	H,-4,7	-8 248 211	
H,-9,2	-9 311 326		11 176 -161	-11 283 -296	-7 169 -118	H,-1,14
-7 182 -191		H,-7,10				-9 180 130
	H,-8,5	-9 223 214	H,-5,3	H,-4,6	H,-2,11	
H,-9,3	4 232 240	-8 325 -291	-11 331 -349	-12 200 -250	-12 229 -158	H,-1,15
-6 308 388	5 219 -207	-7 275 280	-12 275 241		-11 293 257	2 163 -122
				H,-4,5		
H,-9,4	H,-8,3	H,-7,11	H,-5,4	-11 150 107	H,-2,10	H,-1,17
-7 191 -162	-9 253 259	-9 187 162	-13 186 -136		6 150 -121	-2 158 -201
-6 220 -171	-8 283 -319		-12 168 169	H,-4,4		
	-7 197 253	H,-7,12	-11 247 -253	-11 160 -183	H,-2,9	H,0,16
H,-9,5	8 201 -194	-7 186 -75			-13 158 -163	-1 264 -258
7 168 117		-2 183 -134	H,-5,7	H,-4,2	-12 356 359	-2 306 -372
3 186 -119	H,-8,2	0 169 -128	-13 188 -193	10 173 -227		-8 267 252
2 344 362	6 189 -202			-11 444 435	H,-2,5	
1 278 -313	-7 198 250	H,-6,14	H,-5,8		-12 230 203	H,0,6
-4 206 -233	-8 198 -168	-6 188 -144	-11 160 136	H,-3,0		-12 303 381
-5 264 317		-8 210 -156		11 211 208	H,-2,3	-13 182 -269
	H,-3,1		H,-5,9		10 270 240	
H,-9,6	6 380 415	H,-6,13	-12 170 -118	H,-3,1		H,0,4
-2 147 100	7 323 -329	-5 228 204		11 171 239	H,-2,1	10 171 -124
	9 168 -20		H,-5,11	-11 196 -215	-13 170 163	
H,-9,7		H,-6,12	3 208 -212	-12 317 333	-12 183 -107	H,0,2
-4 233 -221	H,-8,0	1 198 199	-11 179 178		11 183 -138	12 252 -253
-7 219 -192	10 164 150			H,-3,2		11 226 200
		H,-6,6	H,-5,12	-13 173 -128	H,-1,1	-14 177 -180
H,-9,9	H,-7,0	-10 260 325	-11 187 123	-12 158 145	13 200 -104	
0 211 193	-11 213 227			10 179 -160	11 164 -164	
-1 201 -230	-10 216 -173	H,-6,5	H,-5,13	11 214 156	-12 159 -147	
-5 205 242	8 183 -105	-12 171 -92	2 252 285			

Some data of the four crystals are given in Table 1. HJBR-1*a* and HJBR-2*a* are synonyms for one of the enantiomers of HJBR-1 and HJBR-2, respectively. The densities were measured by flotation in mixtures of potassium iodide and zinc bromide solutions. The lattice parameters of HJBR-1 and HJBR-1*a* were calculated from series of diffractometer-measured θ values [λ(Mo $K\alpha$)=0·71069 Å], of HJBR-2 from precession films [λ(Mo $K\alpha$)=0·71069 Å], and of HJBR-2*a* from Weissenberg films [λ(Cu $K\alpha$)=1·5405 Å].

Intensity data of the crystals of HJBR-1, HJBR-1*a* and HJBR-2 were collected with a Nonius 3-circle automatic diffractometer by the θ–2θ scan technique and Zr-filtered Mo-radiation for HJBR-1, and by the ω-scan technique and quartz crystal monochromated Mo-radiation for HJBR-1*a* and HJBR-2. Intensities of reflexions of HJBR-1 and HJBR-1*a* were measured in the range $2{\cdot}5° < \theta < 25°$. The scan angle was 1·3°, and the scan speed 1·2°.min^{-1}. For HJBR-2 the range in which intensities of reflexions were measured was $2{\cdot}5° < \theta < 20°$, and the scan speed was lowered to 0·6°.min^{-1} and the scan angle set to 1·1°.

Thus about 3300 independent reflexions were collected from HJBR-1, about 1800 from HJBR-1*a*, and about 2100 from HJBR-2. A reflexion was considered unobserved and was omitted, when the intensity was less than 2·5 times its corresponding estimated standard deviation. Consequently, the numbers of observed reflexions were reduced to 1402, 660 and 1327 for HJBR-1, HJBR-1*a*, and HJBR-2, respectively. These data were corrected for Lorentz and polarization effects, but no corrections for absorption or extinction were made.

Structure determinations

The structures of the three isomers were solved from the corresponding three-dimensional Patterson syntheses by the heavy-atom method. The electron-density maps, based on the bromine atoms only, revealed the positions of all 24 non-hydrogen atoms of the molecules of each structure.

The coordinates of these atoms, as derived from the electron density maps, were subjected to Fourier refinements, during which the conventional R values reduced to 31% (HJBR-1), 20% (HJBR-1*a*), and 21% (HJBR-2). The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

This was followed by full-matrix least-squares refinements, in which positional parameters as well as

Table 3. *Observed and calculated structure factors for* HJBR-1*a*

The columns are h, 10 $|F_o|$, and 10F_c.

H, 0, 0
4 915 903
6 205 261
12 303 -327

H, 0, 1
11 276 -270
9 234 275
8 317 -266
7 575 -625
6 481 -475
5 741 743
4 458 -503
3 1483 1486
2 541 666

H, 0, 2
0 1092 1220
1 1037 1159
3 844 -823
4 853 -850
5 189 -102
7 456 -529
9 193 -211
10 224 176

H, 0, 3
8 585 -493
7 355 -386
6 157 -201
5 303 -289
4 908 -938
3 801 848
2 595 612
1 709 701

H, 0, 4
0 604 -606
1 196 -139
4 608 -557
5 443 467
6 200 215
7 471 415

H, 0, 5
10 328 -242
7 298 283
6 439 380
5 309 283
4 423 384
3 400 -391
2 488 463
1 494 478

H, 0, 6
0 352 327
1 543 -513
2 197 189
3 229 242
5 548 -526
7 297 -322

H, 0, 7
6 228 -150
3 283 293

H, 0, 8
2 458 -342

H, 0, 9
2 261 -294

H, 0, 10
2 239 198

H, 1, 0
6 262 283
5 235 175
3 281 316
1 253 198

H, 1, 7
2 251 -231
3 309 -354
5 257 230
7 211 -116

H, 1, 6
4 218 -134
3 253 -238
2 536 -490
1 184 -205

H, 1, 5
0 356 300
1 274 292
2 681 -685
3 320 340
4 448 443
5 284 -297
6 261 -192

H, 1, 4
11 260 -227
9 195 -112
4 455 454
3 662 657
2 238 -248
1 1027 1072

H, 1, 3
0 888 966
1 805 -858
2 334 347
3 423 446
4 506 -452
5 501 -516
6 362 344
7 266 268
8 275 -327
9 257 -327
10 218 256

H, 1, 2
9 216 -189
8 322 351
7 323 295
6 627 579
5 632 -632
4 852 806
3 501 -471
2 1469 1547
1 1469 1645
0 930 1058

H, 1, 1
2 854 875
3 1135 1118
4 1228 1285
5 685 641
6 307 -306
7 377 -332
8 416 -411
9 352 441

H, 1, 0
9 185 219
8 788 -828
7 335 340
6 602 -645
5 505 -504
4 321 -306
3 2025 2128

H, 2, 0
2 1758 1658
3 1508 1417
4 446 -473
5 851 -850
6 145 -192
7 433 -428
8 185 101
11 298 -339

H, 2, 1
10 192 -96
9 379 -358
7 490 -496
6 184 157
5 357 -354
4 234 -267
3 1617 1505
2 1000 -925
1 816 804

H, 2, 2
0 613 630
1 957 1015
2 477 446
3 686 -683
4 772 791
5 548 1003
7 503 537
8 506 498

H, 2, 3
5 364 346
6 255 279
7 502 481
6 724 714
5 335 320
4 542 -532
3 567 625
2 750 812
1 516 -568
0 478 -442

H, 2, 4
0 581 -606
1 370 -359
2 559 595
3 413 -443
4 765 -774
5 516 -494
6 237 207
7 434 -402
8 216 145
9 312 -263
11 282 -248

H, 2, 5
9 213 -160
8 334 -296
6 227 -202
5 314 -260
4 463 465
3 356 308
2 339 -328
1 158 -157
0 560 570

H, 2, 6
1 357 -303
2 183 -110
3 356 334
6 390 372
7 231 181
10 251 216

H, 2, 7
1 336 278

H, 2, 8
0 382 336
1 214 223
4 229 156

H, 3, 8
5 232 -273

H, 3, 7
0 434 411
4 254 323
5 269 267

H, 3, 6
7 253 -273
6 344 312
5 395 409
4 186 247
2 179 126
1 359 378
0 442 438

H, 3, 5
0 527 565
1 597 -619
3 191 -183
4 272 -254
5 182 -127
6 281 -182
7 200 135

H, 3, 4
12 246 232
10 216 -186
9 291 279
8 302 341
6 509 -491
5 228 263
4 590 587
3 331 -382
2 576 -633
1 452 -514
0 440 -491

H, 3, 3
0 565 613
1 744 785
2 868 858
3 655 647
4 278 -273
5 187 137
6 309 -333
7 295 -239
9 343 -338

H, 3, 2
6 444 -438
5 656 -642
4 425 -427
3 912 923
2 703 -712

H, 3, 1
0 164 263
1 987 -873
2 1006 922
3 505 -433
4 443 421
5 693 664
6 771 768
7 575 -569
8 436 403
10 454 434

H, 3, 0
10 351 352
8 303 -297
7 730 -725
6 972 1067
5 828 725
4 133 -30
3 170 157
2 824 838
1 1009 687

H, 4, 0
1 903 766
2 618 506
3 1160 1133
4 245 273
5 513 519
9 435 -383
13 269 -252

H, 4, 1
10 447 -439
9 236 258
7 178 221
5 439 409
3 152 -196
2 1863 1634
1 819 669
0 131 144

H, 4, 2
0 296 -264
1 848 -814
2 705 -632
3 574 611
4 255 359
5 392 -368
6 1151 1091
8 215 233
10 292 -315
11 352 356

H, 4, 3
11 306 -233
10 214 -239
9 243 -163
7 438 -452
5 333 -345
4 550 -541
2 240 237
1 779 -772
0 637 -708

H, 4, 4
0 192 -160
1 227 201
2 340 365
3 294 -312
4 455 461
5 622 612
6 561 -505
7 437 468
9 386 479

H, 4, 5
9 226 237
8 206 186
7 412 369
6 190 195
5 233 190
4 211 174
3 481 487
2 480 -455
1 294 -279
0 248 140

H, 4, 6
0 215 175
1 364 -404
3 356 -323
4 369 -375
7 241 -222

H, 4, 7
4 313 337
3 257 -318
1 232 152
0 333 266

H, 4, 8
1 275 195

H, 4, 9
0 254 -156

H, 5, 7
1 210 -212
2 224 245

H, 5, 6
7 214 -220
5 227 250
4 279 -276
3 268 -180
2 335 429

H, 5, 5
0 267 272
1 555 553
2 267 291
3 312 360
5 239 250

H, 5, 4
6 565 -597
4 495 544
3 176 -224
2 615 -621
1 349 416
0 546 644

H, 5, 3
1 883 -900
2 530 508
3 952 -948
4 467 431
5 182 221
6 206 123
8 365 373
10 226 119

H, 5, 2
10 318 -321
9 258 -249
8 274 291
7 324 393
6 200 243
5 672 -655
3 887 766
2 592 528
1 519 -511

H, 5, 1
0 612 -569
1 876 765
2 758 -564
3 559 501
4 518 -427
5 190 -254
6 684 -754
7 274 227
8 250 -236
10 353 -349

H, 5, 0
5 476 1045
4 289 -519
3 185 234
2 474 523
1 1192 1007

H, 6, 0
0 172 -140
1 705 -748
2 752 864
3 186 -234
5 958 -971
7 399 424
11 397 381

H, 6, 1
9 253 -370
8 222 240
7 310 -267
6 281 -280
5 510 -471
4 751 -644
3 727 -734
2 904 -740
1 525 477
0 1110 1066

H, 6, 2
1 1299 1178
2 542 519
3 660 577
4 984 834
5 466 -475
8 374 407
13 255 138

H, 6, 3
9 276 279
7 285 354
6 443 419
5 357 444
3 311 316
2 742 724
1 538 -527
0 282 267

H, 6, 4
0 317 349
2 637 -589
3 413 407
4 460 452
5 225 248
6 364 -355
7 253 253
8 250 -224

H, 6, 5
11 336 -107
8 235 -152
7 267 -145
5 321 -354
3 242 -278
1 231 -224
0 405 467

H, 6, 6
2 316 342
3 316 -334
6 304 -260
7 216 -169

H, 6, 7
2 408 -419
0 349 -417

H, 6, 8
1 234 -190
3 257 -249

H, 6, 10
3 254 133

H, 7, 8
1 241 227

H, 7, 7
0 274 -252
3 335 354
5 246 -181

H, 7, 6
6 362 -382
3 208 192
2 538 565

H, 7, 5
1 308 -265
2 261 283
3 421 -477
4 339 -383
5 250 259

H, 7, 4
7 201 -72
5 363 332
4 326 322
3 351 352
2 298 353
1 563 546
0 652 -689

H, 7, 3
1 630 678
2 362 -382
4 276 319
5 416 388
6 313 -375
7 346 -296
8 294 280
10 228 -232

H, 7, 2
8 203 252
7 279 280
5 401 -365
4 722 728
3 329 337
2 661 598
1 972 -845
0 1294 1086

H, 7, 1
1 385 -292
2 423 329

H, 5, 6
0 614 679

H, 7, 1
3 575 472
4 676 687
5 414 -429
7 365 390
8 678 671
9 241 -309

H, 7, 0
9 206 194
8 361 -459
7 370 407
5 490 -492
4 235 -270
3 1356 1125
2 406 240
1 1398 981

H, 8, 0
0 2398 1709
1 524 -403
2 399 256
3 348 263
4 894 -826
8 355 280
11 274 -202

H, 8, 1
8 337 -324
7 359 371
6 300 -345
5 343 -364
4 606 585
3 164 128
2 341 275
1 711 -621
0 536 544

H, 8, 2
1 887 -828
2 228 143
3 708 651
4 256 271
6 207 -224
7 268 374
9 247 225

H, 8, 3
8 272 347
7 413 -429
6 199 -218
4 810 -803
3 461 -440
2 267 -230

H, 8, 4
0 683 691
1 198 -220
2 433 -434
3 293 -279
4 359 339
5 276 -306
6 239 -258
7 338 -278

H, 8, 5
7 317 353
6 279 342
5 337 396
2 254 225
1 238 226

H, 8, 6
0 353 -390
1 475 495
4 238 278
6 258 189

H, 8, 8
2 374 190

H, 9, 6
4 257 317
0 240 273

H, 9, 5
0 248 161
1 210 258
2 415 429
3 324 -383
4 278 -253
8 270 -173

H, 9, 4
3 521 -469
2 206 201
1 478 -557

H, 9, 3
0 652 716

Table 3 (*cont.*)

H,9,3
1 260 271
3 420 -363
4 511 577
5 346 356

H,9,2
8 368 -334
7 256 -222
6 218 -159
5 409 374
4 488 -484
3 205 167
2 704 -645
1 559 545
0 675 631

H,9,1
0 407 -386
2 367 -252
3 752 646
4 422 390
5 310 -343
6 421 -463
7 443 460

H,9,0
8 286 281
6 241 164
5 278 252
4 767 740
3 335 -261
1 977 -790

H,10,C
0 581 358
1 296 -232
2 1133 524
3 354 -291
4 446 372
5 256 -187
7 448 453
10 254 -249

H,10,1
5 234 -287
4 303 194
3 622 643
2 517 -536
1 582 -538
0 384 312

H,10,2
0 445 -401
1 572 -550
2 317 -343
4 379 -404
5 423 -430
6 257 284
7 259 -276

H,10,3
6 381 -374
5 260 250
4 296 308
3 256 -271
2 405 -429
1 452 543
0 302 307

H,10,4
0 361 322
2 372 -350
5 282 360
7 311 321

H,10,5
4 352 -349
3 259 -352
0 407 -477

H,10,7
1 248 -266

H,11,7
0 282 302

H,11,4
6 261 280
4 253 -263
2 215 104
1 437 543

H,11,3
2 565 643
3 372 -357

H,11,2
7 333 -384
6 431 401
3 615 -582
2 219 176
1 312 -372

H,11,1
1 657 583
2 707 -648
5 512 -574

H,11,0
7 313 210
6 654 -652
4 283 273
2 604 -534

H,12,0
0 1014 -965
1 250 23
2 559 -540
5 356 -322
9 287 -338

H,12,1
5 378 -365
4 230 220
3 607 541
2 816 -705
1 262 -330
0 230 384

H,12,2
0 472 434
1 233 134
2 535 485
5 292 196
6 440 460

H,12,3
3 265 -273
2 298 -354
1 296 382

H,12,4
3 243 336
5 377 -404

H,12,5
2 318 298

H,12,6
3 278 253

H,12,6
3 271 272

H,13,4
6 285 295

H,13,3
0 281 359
1 253 346
3 243 225
4 267 -248
5 311 -222

H,13,2
6 281 -192
5 268 363
4 243 -217

H,13,1
0 476 -566
1 251 -188
3 356 -437
6 463 455
5 272 240

H,13,0
4 521 490
3 354 -298
2 381 -306
1 788 -617

H,14,C
5 512 481
7 279 -255

H,14,1
7 282 166
5 280 362
0 297 421

H,14,2
1 468 469
4 368 391

H,15,2
0 416 564

H,15,1
1 271 193
3 275 -179
4 294 -343

H,15,0
3 486 -504
1 288 358

H,16,0
0 510 454
2 316 -345

individual atomic, isotropic thermal parameters were varied. The function minimized was $w(|F_o|-|F_c|)^2$, where $w=1/(A+B|F_o|+C|F_o|^2)$. The coefficients A, B, and C were derived empirically to give very strong and very weak reflexions weights less than 1·0, and the remaining reflexions ($\sim 25<|F_o|<125$) unity weights.

The isotropic refinements ceased at $R=19\%$ (HJBR-1), 18% (HJBR-1*a*), and 17% (HJBR-2). Three cycles of least-squares refinements in which anisotropic thermal parameters were used for the bromine and nitrogen atoms reduced the R values to 12, 11, and 10%.

Difference Fourier maps calculated at this stage showed the approximate positions of the 18 hydrogen atoms of HJBR-1 and HJBR-2. The positions of the hydrogen atoms of HJBR-1*a*, the structure of which is less accurately determined, since it is based on rather few data, were calculated from the positions of the corresponding parent atoms, and checked to fall into positive regions in the difference map.

The contributions of the hydrogen atoms to the scattering were included in the remaining 2 cycles of least-squares refinements, but their positional parameters and temperature factors of 4·0 Å^2 were not varied. The refinements were considered ended, when all parameter shifts were less than $\frac{1}{3}$ of the standard deviations. The final R values are 10·1, 10·8, and 8·6% for HJBR-1, HJBR-1*a*, and HJBR-2, respectively.

Observed and calculated structure factors are listed in Tables 2 to 4. The refined coordinates and thermal factors of the non-hydrogen atoms are listed in Tables 5 to 7, and the approximate coordinates of the hydrogen atoms are listed in Tables 8 to 10.

Most of the calculations were performed on the IBM 7094 computer at NEUCC, Lundtofte, Denmark, using mainly the program system *X-ray* 63 (Stewart, 1964). The cell parameters were refined by means of the least-squares program, described by Liminga (1965). Programs to produce an input tape to, and to process the output tape from, the diffractometer have been written by A. M. Sørensen of this laboratory. The drawings were produced by *ORTEP*, written by Johnson (1965).

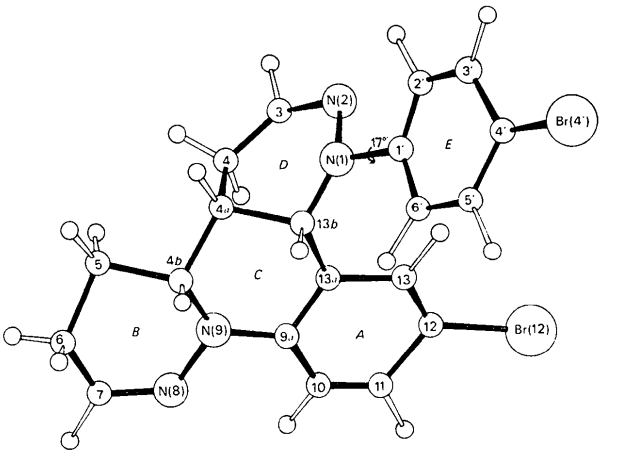

Fig. 2. The molecular structure of HJBR-1*a*.

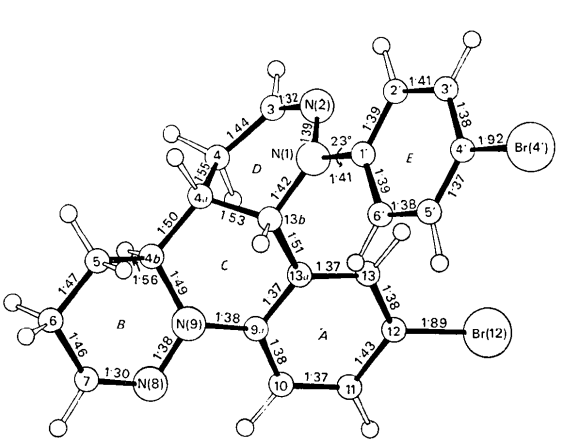

Fig. 3. The molecular structure of HJBR-2.

Table 4. *Observed and calculated structure factors for* HJBR-2

The columns are l, $10F_o$, and $10F_c$.

0,0,L
4 974 1034
6 94 -48
8 830 -846
12 462 412

0,1,L
-3 824 -858
-4 1313 1420
-5 965 -961
-6 115 64
-7 477 -495
-8 1319 1352
-9 282 290
-10 83 -49
-11 279 286
-12 157 105
-13 166 175

0,2,L
-16 175 181
-12 317 -282
-11 121 -95
-9 254 -220
-6 263 -255
-5 639 635
-4 845 863
-2 408 -451

0,3,L
4 211 288
-1 251 -301
-2 1177 1260
-3 138 -96
-5 577 612
-8 252 197
-9 107 115
-10 157 -148
-11 227 -219
-13 185 -183
-14 158 151
-15 112 -107

0,4,L
-12 103 145
-11 177 -153
-10 469 -446
-9 352 322
-8 145 98
-7 174 189
-6 1030 1056
-5 168 -194
-4 249 -227
-3 386 -420
-2 579 -601

0,5,L
-1 284 283
-2 1446 1521
-3 162 -162
-4 594 562
-5 557 524
-6 177 206
-7 243 231
-9 185 -197
-10 440 443
-11 99 63
-13 186 -145
-14 484 -484
-15 256 -263

0,6,L
-17 138 100
-11 214 167
-10 492 444
-9 169 160
-4 318 -344
-3 760 -773
-2 662 704
-1 760 -797
0 330 -342
6 1088 1262
7 235 268
8 132 180

0,7,L
8 221 -204
-2 712 745
-3 614 600
-4 243 -238
-5 691 -691
-6 131 -82
-7 606 -569
-9 247 229
-10 301 -334
-11 443 481
-14 136 85
-16 119 -68

0,8,L
-14 117 -19
-13 276 209
-11 150 -68
-8 229 -204
-6 95 115
-4 386 -373
-3 684 653
-2 155 158
-1 590 598
0 881 890
9 95 34
10 253 -284
16 116 -11

0,9,L
3 452 -488
-4 696 650
-5 362 370
-6 165 165
-7 377 354
-8 552 -520
-9 167 -108
-10 361 -379
-11 227 250

0,10,L
-12 315 -298
-9 172 -111
-8 108 35
-7 190 -174
-6 150 114
-5 444 429
-4 307 250
-1 384 -410
0 811 -814
3 85 -83

0,11,L
14 119 -32
-1 547 -542
-2 105 -123
-3 300 303
-5 181 198
-8 407 344
-9 361 364
-10 119 65
-12 160 -120
-13 324 -289
-15 136 78

0,12,L
-9 216 183
-6 219 210
-5 527 -481
-4 434 -445
-3 421 -379
-1 279 307
0 371 390
2 145 -121
7 365 -366
14 125 -56

0,13,L
-1 429 406
-2 170 -169
-3 235 -232
-4 147 191
-10 186 161
-11 129 132

0,14,L
-11 192 144
-10 161 125
-7 352 -317
-6 141 -51
-4 185 -158
-3 106 -124
-1 143 -131
0 173 199

0,15,L
-2 232 175
-3 335 295
-4 136 88
-5 148 -161
-7 192 -198

0,16,L
-3 150 147
-1 304 283
5 176 189

0,17,L
-3 232 -194
-5 212 164
-7 202, 191

0,18,L
-3 134 -112
-1 216 -214

1,12,L
1 184 -140
0 159 122
-2 135 -92
-3 218 190

1,17,L
-2 116 13
-1 137 -138
1 333 272

1,16,L
7 195 188
3 186 -188
2 113 66
0 144 117
-3 313 -304
-4 159 -102
-7 212 169

1,15,L
-9 124 93
-7 239 147
-6 257 213
-5 163 -144
-2 234 -183
-1 274 281
0 124 -143
1 244 -225
4 190 175
5 121 139
6 149 100
7 116 -86

1,14,L
9 136 138
7 203 -187
6 242 241
4 174 -204
3 311 246
2 224 -171
1 272 -280
0 105 -49
-1 230 -246
-3 139 88
-4 110 142
-5 124 -96
-6 205 162
-9 193 177
-11 133 -129

1,13,L
-9 154 -127
-8 112 -101
-7 170 -192
-6 158 -180
-5 432 359
-4 218 194
-2 418 411
-1 253 -268
2 138 211
5 261 -246
6 300 -295
7 285 254
10 137 114
11 139 -3
12 116 40

1,12,L
11 130 158
10 130 -132
5 108 -79
3 692 -705
2 427 462
-1 406 370
-2 234 -235
-3 335 -375
-4 185 198
-8 253 -227
-9 319 -275
-11 159 152
-13 178 130

1,11,L
-12 126 92
-9 104 39
-8 143 -110
-7 162 139
-6 157 -127
-5 632 -575
-3 146 -124
-2 104 104
-1 314 305
0 487 489
1 124 -150
3 203 154
4 351 -352
5 183 171
7 364 -348
8 154 141
9 256 -248
11 258 362

1,10,L
12 245 178
9 280 251
8 318 -275
6 210 -259
4 132 113
3 571 628
2 287 274
0 354 344
-2 223 232
-3 414 417
-4 623 -608
-5 404 -397
-6 138 -94
-7 154 -142
-8 232 203
-13 149 -122

1,9,L
-14 134 -106
-13 283 -291
-12 190 -93
-11 312 234
-9 248 248
-8 335 309
-7 225 -236
-6 183 -147
-5 380 393
-4 245 251
-3 248 -234
-2 246 -233
-1 479 -483
0 842 -866
1 643 682
3 224 263
4 671 735
5 590 -647
8 123 -164
10 154 -144
11 126 154
16 161 123

1,8,L
12 166 -178
11 319 -360
9 501 -489
8 460 513
7 327 377
5 350 376
4 509 -530
3 189 205
2 644 681
1 302 320
0 226 -233
-1 293 -303
-2 383 -371
-3 421 -421
-4 652 624
-5 266 299
-6 175 159
-7 589 559
-8 320 -317
-11 157 -134
-16 154 145

1,7,L
-15 169 137
-13 153 -187
-12 383 386
-11 377 -339
-10 207 -216
-9 128 -81
-8 185 -198
-6 242 224
-5 302 312
-4 463 -438
-2 805 -802
-1 524 565
0 566 579
1 959 1055
2 196 -193
3 799 -908
4 333 -358
5 410 480
6 222 238
7 256 308
8 188 -233
9 144 113
10 228 -322
12 285 277
13 148 -100

1,6,L
14 184 -152
11 187 144
10 104 127
9 266 283
7 202 -215
6 407 436
5 138 -164
2 913 1022
1 450 449
0 132 -131
-1 258 228
-2 204 209
-3 1031 1048
-4 296 -315
-5 144 -173
-6 236 234
-7 519 -506
-8 337 316
-9 166 138
-10 302 -308
-12 134 -164
-13 196 198
-14 299 273

1,5,L
-13 356 -362
-11 148 146
-9 170 150
-8 201 -207
-7 500 -487
-6 1285 1278
-5 505 483
-4 551 543
-2 1175 1154
-1 438 -455
0 261 256
1 945 1005
2 157 163
3 212 -252
4 347 -415
6 889 1019
7 191 258
8 177 247
10 515 477
12 107 -94
13 138 124

1,4,L
15 117 -67
14 226 151
13 147 133
12 126 -155
11 379 380
10 376 -401
8 109 -88
6 747 -784
5 126 -146
4 98 -95
3 1126 1328
2 1491 1718
1 1243 1353
0 1190 1216
-1 666 644
-2 1298 1307
-3 527 -489
-4 145 -131
-4 175 -131
-4 154 -131
-6 347 -377
-7 288 -269
-8 194 -159
-9 184 -194
-10 802 764
-11 301 295
-12 218 178
-13 428 432
-15 145 -99

1,3,L
-14 177 -117
-13 180 -208
-12 321 300
-10 321 -320
-9 497 485
-7 143 206
-6 860 855
-5 998 -984
-4 284 -263
-3 903 -904
-2 1910 1906
0 1366 1422
1 1353 1473
2 321 -386
3 287 323
4 255 -295
5 153 132
6 661 689
7 856 -985
8 411 -443
9 384 -399
10 452 -457
11 482 515
12 204 -220
13 297 335

1,2,L
15 170 163
14 159 -164
12 176 151
11 165 -182
10 303 282
9 160 -167
8 603 -603
7 490 515
6 390 407
5 920 1024
4 810 886
3 607 694
2 782 -907
0 638 610
-1 1687 1592
-2 379 -349
-3 888 -831
-4 1513 1539
-5 415 423
-7 774 790
-8 560 575
-10 260 -259
-11 222 -256
-13 187 -180
-16 150 -152

1,1,L
-12 361 -325
-10 106 92
-9 104 58
-8 332 363
-7 446 412
-6 590 -552
-5 312 344
-4 699 688
-3 821 802
-2 153 -140
2 631 673
3 350 -378
4 1465 1596
5 498 509
6 482 -487
7 124 -101
9 252 205
10 109 64
12 368 -310
13 109 -106
14 103 129
15 187 -184
16 153 110

1,0,L
12 287 -228
8 560 571
6 594 595
4 897 -903
2 1239 1193
-4 1668 1787
-6 396 428
-8 988 1029
-10 207 -240
-14 173 -152
-16 161 163

2,0,L
-14 216 214
-12 691 -702
-10 784 817
-8 861 918
-6 457 -480
-2 1399 1120
0 3086 2746
2 658 586
4 1414 1349
6 577 -581
8 408 -410
10 163 -211
14 157 121

2,1,L
13 104 123
12 413 371
11 196 -172
10 266 227
9 352 -360
8 933 -898
7 414 -387
6 285 -283
5 553 546
3 907 846
2 210 -159
1 572 555
0 2066 1829
-1 943 690
-2 2586 2057
-3 141 -135
-4 1326 1252
-5 360 -343
-6 326 -317
-7 360 376
-9 380 382
-10 246 -216
-11 288 -311
-12 144 158
-13 140 -166

2,2,L
-13 333 353
-12 112 80
-10 118 -91
-9 126 83
-8 547 -536
-7 89 -113
-6 659 652
-5 1105 1095
-4 244 -223
-3 558 -480
-2 1246 1071
-1 219 193
0 722 620
1 369 360
2 389 -408
4 976 1002
5 372 -421
6 571 584
7 470 -467
8 302 320
11 294 288
13 263 248

2,3,L
12 132 -100
11 182 175
9 263 241
8 124 73
7 519 522
6 301 -324
5 189 -248
4 83 -128
3 973 1068
1 767 -713
0 428 -391
-1 422 430
-3 899 810
-4 460 480
-5 759 727
-6 266 -287
-7 213 -235
-8 337 -352
-9 293 -309
-10 248 284
-11 205 -209
-12 214 235
-13 110 51
-14 294 -317

2,4,L
-12 160 -145
-8 101 -80
-6 643 -656
-5 304 305
-4 694 692
-3 232 213
-2 1206 1175
-1 1240 1173
0 426 -407
1 164 -151
2 124 -161
5 168 182
6 393 -414
7 370 406
9 217 -259
10 593 600
11 211 -198
12 296 -342
13 129 140

2,5,L
13 121 138
12 269 -253
11 262 247
8 155 -107
7 277 -288
6 877 977
5 263 -312
4 987 1121
2 1201 1310
1 125 -143
0 465 -461
-1 423 -393
-2 204 216
-3 311 310
-4 298 -269
-6 628 624
-7 217 -208
-8 622 595

Table 4 (*cont.*)

2,5,L

-10	717	-717

2,6,L

-13	101	-133
-12	156	155
-11	349	-323
-10	136	-158
-7	757	755
-6	1032	1015
-4	118	-125
-3	298	293
-2	1226	1336
-1	232	-208
0	146	155
1	421	-423
2	724	760
3	486	-539
4	99	122
5	750	330
6	345	344
7	265	282
8	312	-353
10	325	-306
11	115	-103
12	168	182
13	205	-184

2,7,L

12	104	-59
9	653	-629
8	218	198
5	422	447
4	284	-311
2	369	399
1	1065	1096
0	135	69
-1	442	-454
-2	1009	-986
-3	1091	1052
-4	280	254
-6	280	-282
-7	220	241
-10	463	478
-12	166	169
-13	115	-133

2,8,L

-8	357	342
-7	388	-369
-6	318	-316
-4	103	-101
-2	66	-67
-1	185	-228
0	628	-659
1	866	885
2	540	569
4	456	501
5	560	-630
6	186	-237
7	260	-296
8	144	-106
9	312	391

2,9,L

11	123	-119

2,10,L

8	441	507
7	300	-309
6	221	239
5	363	391
4	636	-654
2	164	-163
1	109	-119
0	281	275
-1	433	446
-3	185	-198
-4	253	237
-5	359	-357
-6	166	141
-7	207	316
-8	282	-248
-10	196	-149

2,11,L

-9	322	-274
-8	128	-99
-5	190	167
-4	404	438
-3	143	-192
-2	199	-166
-1	329	330
0	434	-443
1	252	274
5	248	-240
7	276	351
8	186	166
9	203	-254

2,12,L

7	320	304
4	225	246
2	268	286
1	113	-151
0	117	-132
-1	634	-606
-3	104	136
-4	120	113
-5	552	528
-6	165	-173
-9	134	124

2,13,L

-5	275	-262
-4	123	-124
-2	126	86
-1	176	-112
0	155	-115
1	235	-245
3	356	351
4	157	192

2,14,L

2	284	275
1	138	-175
-1	408	423
-2	291	-307

2,9,L

-9	175	179
-8	104	143
-7	316	-329
-6	263	-211
-5	165	-187
-4	384	-399
-3	713	742
-2	233	225
-1	192	-164
0	844	838
1	168	-146
3	159	136
4	214	-190
5	92	-79
8	439	-450

3,0,L

-14	503	558
-12	116	111
-8	338	289
-6	632	-609
-2	1136	958
0	689	656
2	1083	-678
4	391	-356
6	284	-293
8	178	-138
10	546	517
12	234	153

3,1,L

11	244	215
9	97	22
8	666	628
7	283	-281
6	856	809
5	557	-533
4	931	-858
3	117	-94
1	465	491
0	680	624
-1	1065	902
-2	598	-448
-3	279	-184
-4	592	542
-6	970	926
-7	598	-562
-8	518	-531
-9	191	-217
-10	344	-354
-11	149	-204

3,2,L

-13	207	180
-12	249	-252
-10	481	480
-9	318	-345
-8	363	-335
-5	524	510
-4	1689	1427
-3	1016	846
-2	763	-657
0	963	-739
1	370	-359
2	1060	1002
3	1077	1017
5	433	-412
7	435	401
8	230	218
9	193	152
11	214	-199
13	250	-249

3,3,L

12	118	-60
11	636	-626
10	645	613
9	266	-269
8	291	268
7	392	401
6	99	83
5	565	594
4	498	489
3	734	734
2	752	-773
1	507	-489
0	826	-761
-1	1160	1001
-2	702	614
-3	172	-137
-6	492	-491
-8	424	429
-9	230	227
-11	312	-301
-12	447	-454
-14	234	195

3,4,L

-10	313	-301
-9	427	418
-8	426	448
-6	785	751
-4	678	-622
-3	200	160
-2	155	-98
-1	281	-227
0	466	-436
1	321	286
2	413	-429
3	190	180
4	757	786
5	269	-271
6	593	590
7	404	-418
8	356	-383
10	326	-344

3,5,L

12	157	138
8	556	-637
7	134	112
5	128	113
4	137	-111
2	573	624
1	453	-410
0	1264	1195
-1	304	263
-2	547	-561
-3	229	-237
-4	601	-560
-5	560	-529
-7	260	240
-9	171	178
-10	100	63
-11	154	-146
-12	367	375
-13	237	215

3,6,L

-8	472	-471
-6	359	-362
-5	164	-131
-4	825	764
-3	760	-717
-2	556	552
-1	104	86
0	214	-193
1	647	641
2	291	320
4	438	-466
6	381	-382
7	151	-154
8	466	512
9	258	-225

3,7,L

11	126	152
10	138	167
9	124	98
5	697	-761
4	363	381
3	129	-104
2	209	-239
0	666	-638
-2	576	576
-3	321	281
-4	239	218
-6	283	-280
-7	432	-463
-8	328	295
-11	231	232
-12	206	-165

3,8,L

-11	188	-171
-10	244	-241
-9	216	-217
-8	103	94
-7	176	-128
-5	100	-76
-4	428	-428
-3	836	832
-2	337	327
1	994	1011
2	396	-394
3	191	-196
5	202	211
8	185	-225
9	338	334

3,9,L

9	391	-386
8	344	378
6	318	332
5	493	496
4	457	-448
1	178	-172
-1	132	141
-4	389	391
-6	346	336
-7	479	504
-8	260	-297
-9	121	-109

3,10,L

-9	332	-336
-7	277	269
-6	224	227
-5	373	370
-4	341	331
-3	228	-208
-2	664	-657
0	801	-823
2	267	279
3	358	-390
4	239	228
6	173	148
7	151	77

3,11,L

7	268	236
6	148	-162
5	162	149
4	175	171
3	224	228
2	144	104
1	391	432
0	232	-250
-1	617	-616
-4	204	-224
-6	205	-209
-8	228	214

3,12,L

-5	373	-355
-4	153	-68
-3	132	76
0	130	-135
2	138	-159
3	518	478
4	109	-117
5	119	-141

3,13,L

3	309	-337
2	250	207
-1	305	294
-2	229	-219
-4	176	-137

4,0,L

-12	235	212
-10	130	-186
-8	328	-348
-6	544	592
-2	499	-485
0	402	-378
2	412	-392
4	154	102
6	866	725
8	580	529
10	556	-478
12	257	-264

4,1,L

12	120	-121
11	436	-445
10	192	-135
8	195	-191
7	561	493
6	198	-187
5	234	212
4	436	415
2	977	914
1	796	-753
0	918	-906
-1	564	-470
-2	1457	1179
-3	241	-193
-4	523	472
-5	628	551
-6	231	-222
-10	390	429
-10	395	429

4,2,L

-13	184	-157
-12	281	-297
-9	260	263
-8	415	427
-6	754	-733
-3	118	-156
-2	1227	1012
-1	132	-184
0	363	-310
1	602	574
2	139	-100
3	1057	1036
4	617	613
5	335	325
6	539	-510
7	347	-360
9	555	-540
11	196	-163

4,3,L

10	119	179
9	160	186
7	366	-336
6	416	423
5	500	-504
4	267	267
3	435	437
2	645	-639
1	455	459
0	165	171
-2	496	-433
-3	285	-236
-4	875	-821
-5	941	-841
-6	146	136
-7	198	-235
-8	322	341
-10	241	-290
-12	128	162

4,4,L

-12	169	222
-10	203	202
-8	394	-405
-4	209	78
-3	670	582
-2	781	-695
-1	377	388
0	885	821
1	173	-162
2	382	395
3	107	-155
4	484	-510
5	102	41
9	193	213
10	160	-166

4,5,L

11	119	-114
9	140	-125
8	224	191
7	115	110
6	313	-332
4	594	-597
2	98	-89
1	389	360
-1	104	-87
-2	487	443
-3	311	-286
-4	784	786
-5	148	138
-6	558	-545
-8	545	-567
-9	201	-189
-10	279	266

4,6,L

-9	157	102
-8	239	244
-7	416	-436
-6	257	-223
-5	124	70
-4	301	278
-3	468	442
-2	591	582
-1	192	-153
0	609	-616
1	120	108
2	388	-432
3	97	-121
4	368	411
5	494	-498
6	130	189
9	366	358

4,7,L

8	151	-147
7	241	280
6	106	75
5	349	371
2	214	-242
0	228	267
-2	170	170
-3	509	501
-4	279	-320
-5	247	242
-7	159	179
-8	226	205
-9	222	222
-10	378	-365
-11	285	-296

4,8,L

-10	151	-189
-8	201	-220
-7	330	348
-6	274	309
-5	312	-300
-3	630	-656
-2	230	-216
-1	297	272
3	225	244
4	184	-237
5	414	457
6	284	267
7	193	-167
8	154	131
9	337	-339

4,9,L

7	186	179
6	169	-248
5	243	-276
4	242	281
3	377	-359
2	498	518
1	423	404
0	312	-325
-1	182	131
-2	316	-311
-3	296	-294
-6	229	-231
-8	184	196
-9	170	-210

4,10,L

-7	247	-283
-5	304	311
-4	214	-212
-2	620	614
-1	171	-158
1	162	94
3	183	219
6	351	-349

4,11,L

4	231	-223
3	299	317
2	394	-432
0	119	151
-1	124	120
-2	134	125
-3	335	338
-4	146	-176
-5	484	-494
-6	116	111

4,12,L

-1	469	470

5,10,L

2	261	-269
1	151	-154
0	163	210
-2	439	447
-3	150	160

5,9,L

-6	307	-310
-3	250	235
-2	466	467
-1	427	-416
5	229	-218

5,8,L

5	230	-215
4	274	263
3	328	-347
2	254	269
1	569	577
0	222	-253
-1	129	98
-2	126	154
-3	289	-300
-5	190	198
-6	224	-253
-7	237	-245

5,7,L

-8	148	-198
-7	279	306
-5	489	-488
-4	336	317
-3	193	-196
-2	382	-420
-1	181	177
0	232	272
1	208	214
2	104	26
3	206	234
4	216	-233
7	250	-245

5,6,L

8	370	-394
5	301	337
4	193	182
3	374	383
1	454	-456
0	470	494
-1	316	-342
-2	108	53
-3	209	156
-4	789	-793
-8	175	181
-9	263	302

5,5,L

-8	465	540
-7	132	-78
-6	147	-133
-5	303	293
-3	265	306
-2	331	-291
-1	187	-126
0	478	-496
2	238	-290
4	642	678
5	155	-188
6	213	238
7	106	18
9	139	135

5,4,L

8	565	561
7	173	161
6	140	142
5	298	325
4	135	-183
3	286	289
2	236	-267
1	203	-171
0	714	-738
-1	256	-242
-2	193	192
-3	146	-174
-4	661	678
-6	556	-493
-7	153	153
-8	207	-249
-10	136	165

5,3,L

-9	253	-253
-8	395	-399
-6	298	271
-5	369	356
-4	256	209
-3	208	219
-2	385	-383
-1	392	358
0	583	543
1	479	-500
3	481	-486
4	499	-495
6	125	-130
7	247	242
9	432	415

5,2,L

10	142	-197
9	431	414
6	287	288

Table 4 (*cont.*)

	5,2,L			-4	254	-250		-4	155	-72		0	159	184		7	309	356		4	509	564		6,7,L	
	5	475	-457		-2	980	919		-6	432	-440		-1	259	331						5	220	-194		3	319	-312
	3	109	-139		-1	334	-256		-10	371	427		-3	218	219		6,3,L								1	136	68
	2	688	-710		1	248	219						-4	150	-99		5	175	232		6,5,L				-1	187	254
	1	113	179		2	220	-212		6,0,L				-5	122	-44		3	258	317		4	154	-224		-3	119	91
	0	413	449		3	301	276		-6	261	-306		-6	250	357		2	223	221		2	174	182		-4	224	272
	-1	616	590		5	107	-66		-2	783	816		-9	158	-135		1	195	187		1	118	-150				
	-2	342	345		6	555	-518		0	171	209						0	129	-203		0	560	616		6,8,L		
	-3	151	178		8	136	-102		2	420	-466		6,2,L				-3	283	-342		-2	284	-255		-1	451	-406
	-4	354	-313		9	283	241		4	250	-256		-8	222	-313		-4	915	904		-3	215	186				
	-5	503	-461		10	291	225		6	132	-11		-7	261	241		-5	314	267		-4	162	-244		7,3,L		
	-7	230	-271										-6	273	301		-6	228	-208						-1	175	244
	-10	381	-433		5,0,L				6,1,L				-5	249	261		-7	128	164		6,6,L						
					10	252	-243		7	118	-70		-2	400	-421						-3	203	-163		7,2,L		
	5,1,L				8	240	-261		6	470	451		-1	569	-546		6,4,L				-1	329	339		1	168	218
	-11	151	133		6	817	-730		5	218	-259		0	237	260		-8	394	457		0	240	239				
	-10	121	93		4	251	270		4	133	77		1	494	-557		-5	198	-167		1	117	153				
	-9	153	180		2	928	934		3	224	-254		2	246	286		-4	721	-712		3	113	-110				
	-8	346	257		0	165	174		2	410	-465		3	307	-325		0	306	-312		4	306	-349				
	-6	627	-660		-2	547	-485		1	171	-190		4	172	-193		1	231	263								

Table 5. *Fractional atomic coordinates and thermal parameters* for HJBR-1

The anisotropic thermal parameters are of the form:

$$T=\exp\left[-\tfrac{1}{4}\left(B_{11}h^2a^{*2}+\ldots 2B_{23}klb^*c^*\right)\right].$$

	x	*y*	*z*
Br(12)	1·3936 (2)	0·1058 (2)	0·4354 (2)
Br(4′)	1·5664 (2)	0·8316 (2)	0·2808 (2)
N(1)	1·1272 (14)	0·5060 (15)	0·2417 (11)
N(2)	1·0840 (16)	0·4306 (15)	0·1675 (12)
N(8)	0·8863 (14)	0·3507 (16)	0·4576 (9)
N(9)	0·9393 (11)	0·3891 (15)	0·4006 (8)
C(3)	0·9750 (17)	0·3786 (18)	0·1501 (12)
C(4)	0·9063 (17)	0·3931 (20)	0·2076 (13)
C(4*a*)	0·9459 (16)	0·5047 (19)	0·2718 (13)
C(4*b*)	0·9026 (16)	0·5067 (19)	0·3454 (13)
C(5)	0·7673 (18)	0·5183 (21)	0·3130 (14)
C(6)	0·7279 (16)	0·5119 (20)	0·3891 (13)
C(7)	0·7906 (19)	0·4136 (21)	0·4547 (15)
C(9*a*)	1·0448 (16)	0·3255 (21)	0·4092 (12)
C(10)	1·0763 (16)	0·2112 (19)	0·4595 (13)
C(11)	1·1815 (18)	0·1514 (21)	0·4721 (14)
C(12)	1·2507 (16)	0·1970 (19)	0·4207 (12)
C(13)	1·2199 (15)	0·3073 (18)	0·3739 (12)
C(13*a*)	1·1151 (15)	0·3747 (17)	0·3636 (11)
C(13*b*)	1·0856 (15)	0·5009 (18)	0·3125 (12)
C(1′)	1·2404 (15)	0·5741 (17)	0·2553 (13)
C(2′)	1·2574 (16)	0·6046 (19)	0·1786 (12)
C(3′)	1·3518 (16)	0·6786 (19)	0·1905 (12)
C(4′)	1·4235 (17)	0·7217 (19)	0·2658 (14)
C(5′)	1·4163 (18)	0·6903 (20)	0·3427 (14)
C(6′)	1·3146 (17)	0·6166 (20)	0·3324 (13)

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Br(12)	5·4	6·3	8·3	2·8	1·7	0·8
Br(4′)	5·0	5·5	10·7	−1·2	3·4	0·7
N(1)	6·1	2·7	3·6	0·7	1·0	1·4
N(2)	7·8	2·6	5·8	0·0	2·8	−2·6
N(8)	6·6	5·1	2·7	0·0	2·8	1·4
N(9)	3·1	4·5	1·3	−0·3	−0·3	1·4

	B(Å²)		*B*(Å²)
C(3)	3·5	C(12)	4·0
C(4)	4·6	C(13)	3·7
C(4*a*)	3·9	C(13*a*)	3·1
C(4*b*)	3·8	C(13*b*)	3·1
C(5)	5·1	C(1′)	3·0
C(6)	4·3	C(2′)	3·8
C(7)	4·9	C(3′)	3·7
C(9*a*)	3·9	C(4′)	4·2
C(10)	4·1	C(5′)	4·8
C(11)	5·2	C(6′)	4·6

Table 6. *Fractional atomic coordinates and thermal parameters for* HJBR-1*a*

	x	*y*	*z*
Br(12)	0·4622 (5)	0·7981 (3)	0·6912 (7)
Br(4′)	0·7852 (6)	1·0650 (3)	0·1313 (8)
N(1)	0·601 (3)	0·698 (2)	0·165 (4)
N(2)	0·496 (3)	0·695 (3)	0·103 (5)
N(8)	0·644 (3)	0·391 (2)	0·510 (4)
N(9)	0·639 (3)	0·471 (2)	0·417 (4)
C(3)	0·476 (5)	0·609 (3)	0·101 (6)
C(4)	0·509 (4)	0·528 (4)	0·152 (7)
C(4*a*)	0·618 (3)	0·535 (2)	0·186 (5)
C(4*b*)	0·673 (3)	0·468 (2)	0·282 (4)
C(5)	0·660 (4)	0·372 (3)	0·207 (5)
C(6)	0·708 (4)	0·307 (3)	0·320 (5)
C(7)	0·682 (4)	0·314 (3)	0·456 (6)
C(9*a*)	0·597 (3)	0·543 (2)	0·492 (4)
C(10)	0·563 (3)	0·546 (2)	0·626 (5)
C(11)	0·531 (4)	0·610 (3)	0·677 (5)
C(12)	0·515 (3)	0·697 (3)	0·594 (5)
C(13)	0·546 (5)	0·699 (4)	0·469 (6)
C(13*a*)	0·595 (3)	0·623 (2)	0·401 (4)
C(13*b*)	0·639 (3)	0·634 (2)	0·255 (4)
C(1′)	0·634 (5)	0·791 (4)	0·167 (7)
C(2′)	0·607 (3)	0·840 (2)	0·058 (5)
C(3′)	0·648 (4)	0·919 (3)	0·041 (5)
C(4′)	0·725 (4)	0·954 (2)	0·142 (5)
C(5′)	0·753 (4)	0·889 (4)	0·246 (6)
C(6′)	0·719 (3)	0·810 (2)	0·253 (4)

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Br(12)	6·1	3·7	7·8	−1·0	1·4	−2·3
Br(4′)	8·5	1·9	8·2	−1·4	1·0	0·4

	B (Å²)		*B* (Å²)
N(1)	3·3	C(10)	1·8
N(2)	5·9	C(11)	3·2
N(8)	3·7	C(12)	4·0
N(9)	3·1	C(13)	5·3
C(3)	5·8	C(13*a*)	1·7
C(4)	6·0	C(13*b*)	1·6
C(4*a*)	2·4	C(1′)	6·0
C(4*b*)	1·6	C(2′)	1·9
C(5)	3·5	C(3′)	3·9
C(6)	3·7	C(4′)	2·9
C(7)	4·4	C(5′)	4·6
C(9*a*)	1·7	C(6′)	1·1

Description of the structures

The geometry of the molecules is shown in Figs. 1 to 3. Each of the molecules has three asymmetric car-

Table 7. *Fractional atomic coordinates and thermal parameters for* HJBR-2

	x	*y*	*z*
Br(12)	0·2787 (2)	1·2651 (1)	0·3383 (1)
Br(4′)	−0·6357 (2)	1·1472 (1)	0·0869 (1)
N(1)	0·0297 (15)	0·9527 (8)	0·2216 (8)
N(2)	0·1626 (18)	0·9432 (8)	0·1647 (7)
N(8)	0·2118 (16)	0·8878 (9)	0·5513 (9)
N(9)	0·1869 (14)	0·9016 (8)	0·4662 (9)
C(3)	0·3139 (23)	0·9054 (11)	0·1931 (10)
C(4)	0·3547 (20)	0·8685 (9)	0·2743 (9)
C(4*a*)	0·1788 (18)	0·8519 (9)	0·3192 (9)
C(4*b*)	0·2134 (19)	0·8273 (9)	0·4093 (10)
C(5)	0·0833 (22)	0·7549 (12)	0·4369 (11)
C(6)	0·1280 (23)	0·7330 (12)	0·5252 (11)
C(7)	0·1835 (25)	0·8086 (14)	0·5756 (12)
C(9*a*)	0·2138 (17)	0·9856 (9)	0·4405 (8)
C(10)	0·2893 (20)	1·0486 (10)	0·4923 (9)
C(11)	0·3105 (19)	1·1314 (10)	0·4633 (10)
C(12)	0·2521 (19)	1·1512 (10)	0·3787 (9)
C(13)	0·1769 (19)	1·0870 (9)	0·3276 (9)
C(13*a*)	0·1588 (17)	1·0055 (8)	0·3599 (8)
C(13*b*)	0·0665 (19)	0·9350 (9)	0·3080 (9)
C(1′)	−0·1252 (20)	0·9965 (9)	0·1890 (9)
C(2′)	−0·1644 (21)	1·0000 (10)	0·1031 (10)
C(3′)	−0·3191 (20)	1·0454 (10)	0·0723 (9)
C(4′)	−0·4234 (20)	1·0863 (9)	0·1286 (10)
C(5′)	−0·3911 (20)	1·0804 (10)	0·2133 (10)
C(6′)	−0·2370 (20)	1·0375 (10)	0·2434 (9)

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Br(12)	4·7	4·1	8·9	−1·3	2·5	−0·2
Br(4′)	3·5	6·3	8·3	0·2	−1·6	1·9
N(1)	1·4	4·6	4·0	1·3	1·2	0·3
N(2)	4·3	4·7	2·7	−0·2	−0·1	−0·6
N(8)	2·5	6·2	3·3	0·1	0·2	0·7
N(9)	1·9	4·8	3·5	−0·1	0·1	−0·2

	B (Å²)		*B* (Å²)
C(3)	3·8	C(12)	3·2
C(4)	3·3	C(13)	2·8
C(4*a*)	2·9	C(13*a*)	1·9
C(4*b*)	2·7	C(13*b*)	2·8
C(5)	5·0	C(1′)	2·7
C(6)	4·9	C(2′)	3·9
C(7)	5·3	C(3′)	3·2
C(9*a*)	2·0	C(4′)	3·3
C(10)	3·6	C(5′)	3·5
C(11)	3·6	C(6′)	3·3

Table 8. *Approximate coordinates for the hydrogen atoms of* HJBR-1

	x	*y*	*z*
H(3)	0·943	0·326	0·093
H(41)	0·913	0·305	0·245
H(42)	0·816	0·409	0·167
H(4*a*)	0·913	0·597	0·233
H(4*b*)	0·937	0·589	0·388
H(51)	0·725	0·436	0·268
H(52)	0·736	0·608	0·275
H(61)	0·633	0·495	0·370
H(62)	0·744	0·609	0·423
H(7)	0·747	0·380	0·496
H(10)	1·021	0·170	0·491
H(11)	1·214	0·068	0·519
H(13)	1·279	0·346	0·340
H(13*b*)	1·119	0·582	0·356
H(2′)	1·197	0·577	0·111
H(3′)	1·379	0·698	0·134
H(5′)	1·476	0·715	0·408
H(6′)	1·284	0·588	0·385

Table 9. *Approximate coordinates for the hydrogen atoms of* HJBR-1*a*

	x	*y*	*z*
H(3)	0·410	0·598	0·035
H(41)	0·495	0·476	0·077
H(42)	0·467	0·512	0·250
H(4*a*)	0·655	0·535	0·083
H(4*b*)	0·754	0·486	0·286
H(51)	0·701	0·368	0·105
H(52)	0·581	0·356	0·185
H(61)	0·790	0·318	0·317
H(62)	0·693	0·240	0·288
H(7)	0·691	0·256	0·530
H(10)	0·563	0·485	0·688
H(11)	0·505	0·609	0·794
H(13)	0·533	0·760	0·408
H(13*b*)	0·722	0·644	0·266
H(2′)	0·551	0·814	−0·020
H(3′)	0·631	0·963	−0·048
H(5′)	0·813	0·910	0·332
H(6′)	0·757	0·757	0·319

Table 10. *Approximate coordinates for the hydrogen atoms of* HJBR-2

	x	*y*	*z*
H(3)	0·423	0·900	0·150
H(41)	0·438	0·914	0·314
H(42)	0·433	0·809	0·271
H(4*a*)	0·112	0·797	0·286
H(4*b*)	0·352	0·804	0·419
H(51)	0·091	0·697	0·398
H(52)	−0·056	0·777	0·430
H(61)	0·241	0·687	0·528
H(62)	0·016	0·701	0·552
H(7)	0·198	0·799	0·643
H(10)	0·325	1·030	0·557
H(11)	0·377	1·182	0·501
H(13)	0·139	1·102	0·262
H(13*b*)	−0·060	0·925	0·336
H(2′)	−0·074	0·969	0·061
H(3′)	−0·356	1·049	0·003
H(5′)	−0·476	1·110	0·262
H(6′)	−0·208	1·033	0·308

bon atoms, *e.g.* C(4*b*), C(4*a*), and C(13*b*). The racemates HJBR-1 and HJBR-2 differ only in the chirality at C(4*b*). The crystals of HJBR-1*a* are composed of one of the enantiomers of HJBR-1. No attempts were made to determine the absolute configuration of HJBR-1*a*.

The ring *A* of the condensed ring system of each compound and the rings *E* are aromatic and planar within the experimental error. Each of the rings *B*, *C*, and *D* of the ring systems is 'cyclohexene-like' and half-chair conformations (4 atoms in a plane) were expected. But probably due to the nearly plane trigonal configurations of the nitrogen atoms N(1) and N(9), sofa forms (5 atoms in a plane) or something between sofa and half-chair forms actually were found in most cases. The conformations of the rings are characterized by the valency angles (Table 13) and the torsional angles of the individual bonds, which are given in Table 11.

A comparison of the sequences of the torsional angles shows that the *B* rings of HJBR-1 and HJBR-1*a*

have something between sofa and half-chair conformations with C(5) as the top atoms, while the *B* ring of HJBR-2 has nearly sofa conformation with C(4*b*) as top atom. The *C* rings of HJBR-1 and HJBR-1*a* are sofa forms with C(4*a*) as top atoms, while the *C* ring of HJBR-2 is nearly a boat form with C(13*b*) and N(9) as top and end atoms. The conformation of the *D* ring is almost the same in the three molecules, *i.e.* nearly half-chair conformation with C(4*a*) as the top atom.

The result of the analysis of the conformations is that the ring systems of HJBR-1 and HJBR-1*a* have almost identical conformations, whereas the opposite chirality at C(4*b*) of HJBR-2 has caused a change in the conformations of the rings *B* and *C* of this mol-

Table 11. *Torsional angles* (°) *of the rings, B, C and D of* HJBR-1, HJBR-1*a*, *and* HJBR-2

The theoretical values given are those of Hendrickson (1961).

Torsional angles	HJBR-1	HJBR-1*a*	HJBR-2	Theoretical values for a		
Ring *B*				sofa form	half-chair form	
8–7	7	6	0	0	0	
7–6	+12	+22	+6	+30	+19	
6–5	−41	−47	+36	−60	−56	
5–4*b*	+53	+50	−59	+60	+75	
4*b*–9	−38	−31	+56	−30	−56	
9–8	8	0	−27	0	+19	
Ring *C*				sofa form	half-chair form	boat form
9*a*–9	1	1	+39	0	0	+60
9–4*b*	+30	+28	−28	+30	+19	−60
4*b*–4*a*	−60	−60	−19	−60	−56	0
4*a*–13*b*	+62	+59	+54	+60	+75	+60
13*b*–13*a*	−36	−36	−47	−30	−56	−60
13*a*–9*a*	6	6	3	0	+19	0
Ring *D*				sofa form	half-chair form	
2–3	3	−13	4	0	0	
3–4	+19	+24	+18	+30	+19	
4–4*a*	−44	−38	−47	−60	−56	
4*a*–13*b*	+56	+53	+59	+60	+75	
13*b*–1	−46	−49	−43	−30	−56	
1–2	+20	+27	+9	0	+19	

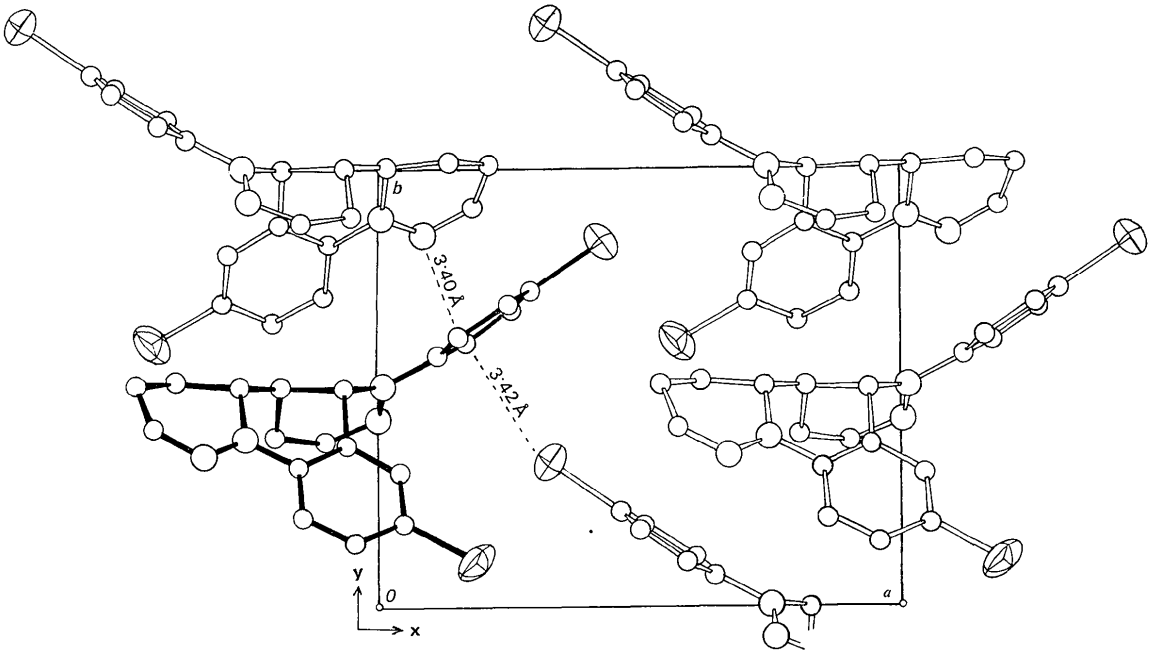

Fig. 4. The structure of HJBR-1 viewed along the *c** axis.

ecule. In addition the nitrogen atom, N(9), of HJBR-2 has not quite plane trigonal configuration. The distance of this atom to the plane through the three neighbouring atoms is 0·19 Å.

The conformations of the molecules might also be expected to differ owing to rotation of the phenyl groups around the N(1)–C(1′) bonds, but actually the dihedral angles are about 20° in all the three molecules (*cf.* Figs. 1 to 3).

The bond lengths and valency angles of the molecules are given in Tables 12 and 13. The values of HJBR-1*a* will not be discussed, because of the low accuracy of this structure. Good agreement was found between the lengths of corresponding bonds of HJBR-1 and HJBR-2 (*cf.* Figs. 1 and 3). The lengths of the four C=N bonds vary between 1·30 and 1·36 Å, and of the four N–N bonds between 1·37 and 1·39 Å. The lengths of the four N–C(phenyl) bonds, on the other hand, vary between 1·39 and 1·49 Å, the longest bond being the N(1)–C(1′) bond of HJBR-1. The intramolecular, non-bonded distance between C(13) and C(6′) is also greater in HJBR-1 (3·48 Å) than the corresponding distance in HJBR-2 (3·37 Å).

Table 12. *Bond lengths of* HJBR-1, HJBR-2 *and* HJBR-1*a*

The estimated standard deviations are in parentheses and refer to the last decimal positions.

		HJBR-1	HJBR-2	HJBR-1*a*
i	*j*	*L*(*ij*)	*L*(*ij*)	*L*(*ij*)
Br(12)	C(12)	1·91 (2) Å	1·89 (1) Å	1·90 (5) Å
Br(4′)	C(4′)	2·01 (2)	1·92 (1)	1·82 (4)
N(9)	N(8)	1·37 (2)	1·38 (2)	1·49 (5)
N(1)	N(2)	1·38 (2)	1·39 (1)	1·49 (6)
N(8)	C(7)	1·32 (3)	1·30 (2)	1·35 (7)
N(2)	C(3)	1·36 (2)	1·32 (2)	1·30 (7)
N(9)	C(9*a*)	1·40 (2)	1·38 (1)	1·39 (5)
N(1)	C(1′)	1·49 (2)	1·41 (1)	1·45 (7)
N(9)	C(4*b*)	1·47 (2)	1·49 (2)	1·35 (5)
N(1)	C(13*b*)	1·42 (3)	1·42 (2)	1·38 (5)
C(7)	C(6)	1·47 (2)	1·46 (2)	1·34 (7)
C(3)	C(4)	1·47 (3)	1·44 (2)	1·38 (8)
C(6)	C(5)	1·49 (3)	1·47 (2)	1·57 (7)
C(4)	C(4*a*)	1·50 (3)	1·55 (2)	1·46 (8)
C(5)	C(4*b*)	1·55 (2)	1·56 (2)	1·61 (6)
C(4*a*)	C(4*b*)	1·48 (3)	1·50 (2)	1·53 (6)
C(4*a*)	C(13*b*)	1·60 (2)	1·53 (2)	1·64 (5)
C(13*b*)	C(13*a*)	1·50 (2)	1·51 (1)	1·49 (6)
C(9*a*)	C(10)	1·39 (3)	1·38 (2)	1·34 (6)
C(10)	C(11)	1·37 (3)	1·37 (2)	1·16 (6)
C(11)	C(12)	1·46 (3)	1·43 (2)	1·53 (7)
C(12)	C(13)	1·33 (2)	1·38 (2)	1·24 (8)
C(13)	C(13*a*)	1·41 (2)	1·37 (2)	1·46 (7)
C(13*a*)	C(9*a*)	1·41 (3)	1·37 (1)	1·48 (6)
C(1′)	C(2′)	1·38 (2)	1·39 (2)	1·31 (8)
C(2′)	C(3′)	1·33 (2)	1·41 (2)	1·32 (6)
C(3′)	C(4′)	1·31 (2)	1·38 (2)	1·49 (7)
C(4′)	C(5′)	1·33 (3)	1·37 (2)	1·43 (7)
C(5′)	C(6′)	1·41 (3)	1·38 (2)	1·27 (7)
C(6′)	C(1′)	1·34 (2)	1·39 (2)	1·41 (8)
Non-bonded distances				
C(13)	C(6′)	3·49	3·37	3·47

Table 13. *Valency angles of* HJBR-1, HJBR-2 *and* HJBR-1*a*

The estimated standard deviations are in parentheses.

i	*j*	*k*	HJBR-1 Angle (*ijk*)	HJBR-2 Angle (*ijk*)	HJBR-1*a* Angle (*ijk*)
N(8)	N(9)	C(9*a*)	116 (1)°	115 (1)°	110 (3)°
N(8)	N(9)	C(4*b*)	123 (1)	118 (1)	121 (3)
C(9*a*)	N(9)	C(4*b*)	120 (1)	121 (1)	129 (3)
C(7)	N(8)	N(9)	118 (1)	115 (1)	119 (4)
N(1)	N(2)	C(3)	116 (2)	116 (1)	103 (4)
N(2)	N(1)	C(13*b*)	124 (1)	121 (1)	123 (3)
N(2)	N(1)	C(1′)	116 (1)	114 (1)	108 (4)
C(13*b*)	N(1)	C(1′)	118 (1)	124 (1)	123 (4)
N(8)	C(7)	C(6)	126 (2)	129 (1)	122 (5)
C(7)	C(6)	C(5)	114 (1)	113 (1)	120 (4)
C(6)	C(5)	C(4*b*)	109 (1)	109 (1)	103 (3)
C(5)	C(4*b*)	N(9)	109 (1)	106 (1)	114 (3)
C(4*a*)	C(4*b*)	N(9)	112 (1)	112 (1)	112 (3)
C(5)	C(4*b*)	C(4*a*)	111 (1)	112 (1)	116 (3)
C(4*b*)	C(4*a*)	C(4)	118 (1)	113 (1)	123 (4)
C(4*b*)	C(4*a*)	C(13*b*)	107 (1)	113 (1)	106 (3)
C(4)	C(4*a*)	C(13*b*)	108 (1)	106 (1)	109 (4)
C(4*a*)	C(4)	C(3)	114 (1)	110 (1)	108 (5)
C(4)	C(3)	N(2)	124 (1)	128 (1)	143 (6)
N(1)	C(13*b*)	C(13*a*)	114 (1)	117 (1)	120 (3)
N(1)	C(13*b*)	C(4*a*)	107 (1)	110 (1)	109 (3)
C(4*a*)	C(13*b*)	C(13*a*)	105 (1)	108 (1)	101 (3)
Br(12)	C(12)	C(11)	117 (1)	119 (1)	118 (3)
Br(12)	C(12)	C(13)	123 (1)	121 (1)	123 (4)
C(11)	C(12)	C(13)	120 (1)	120 (1)	118 (5)
C(10)	C(11)	C(12)	118 (1)	119 (1)	123 (5)
C(11)	C(10)	C(9*a*)	120 (2)	120 (1)	123 (4)
N(9)	C(9*a*)	C(10)	120 (2)	123 (1)	129 (4)
N(9)	C(9*a*)	C(13*a*)	119 (1)	117 (1)	110 (3)
C(10)	C(9*a*)	C(13*a*)	121 (1)	120 (1)	121 (3)
C(9*a*)	C(13*a*)	C(13)	117 (1)	122 (1)	113 (4)
C(9*a*)	C(13*a*)	C(13*b*)	122 (1)	117 (1)	128 (3)
C(13)	C(13*a*)	C(13*b*)	121 (1)	120 (1)	119 (4)
C(12)	C(13)	C(13*a*)	123 (2)	119 (1)	122 (5)
N(1)	C(1′)	C(2′)	113 (1)	120 (1)	117 (5)
N(1)	C(1′)	C(6′)	125 (2)	119 (1)	116 (5)
C(2′)	C(1′)	C(6′)	121 (1)	120 (1)	123 (5)
C(1′)	C(2′)	C(3′)	113 (1)	119 (1)	120 (5)
C(2′)	C(3′)	C(4′)	126 (2)	119 (1)	121 (4)
C(3′)	C(4′)	C(5′)	125 (2)	123 (1)	112 (4)
C(4′)	C(5′)	C(6′)	111 (1)	118 (1)	125 (5)
C(1′)	C(6′)	C(5′)	124 (2)	121 (1)	117 (4)
Br(4′)	C(4′)	C(3′)	124 (1)	119 (1)	125 (3)
Br(4′)	C(4′)	C(5′)	111 (1)	118 (1)	123 (4)

The agreement between corresponding valency angles of HJBR-1 and HJBR-2 also seems reasonable, with the exception of the valency angles of one of the phenyl groups (ring *E* in Figs. 1 and 3). Apparently a deformation of the benzene ring of HJBR-1 has occurred such that the oppositely situated angles C(1′)–C(2′)–C(3′) and C(4′)–C(5′)–C(6′) have become rather small (113 and 111° respectively). In addition the differences between the angles N(1)–C(1′)–C(6′) and N(1)–C(1′)–C(2′) (125 and 113°) and between Br(4′)–C(4′)–C(3′) and Br(4′)–C(4′)–C(5′) (124 and 111°) of HJBR-1 are significant.

The reason for these deformations of the HJBR-1 molecule might be steric repulsion between the molecules. Some rather short intermolecular distances were found from one of the phenyl carbon atoms to some atoms of the neighbouring molecules (*cf.* Fig. 4).

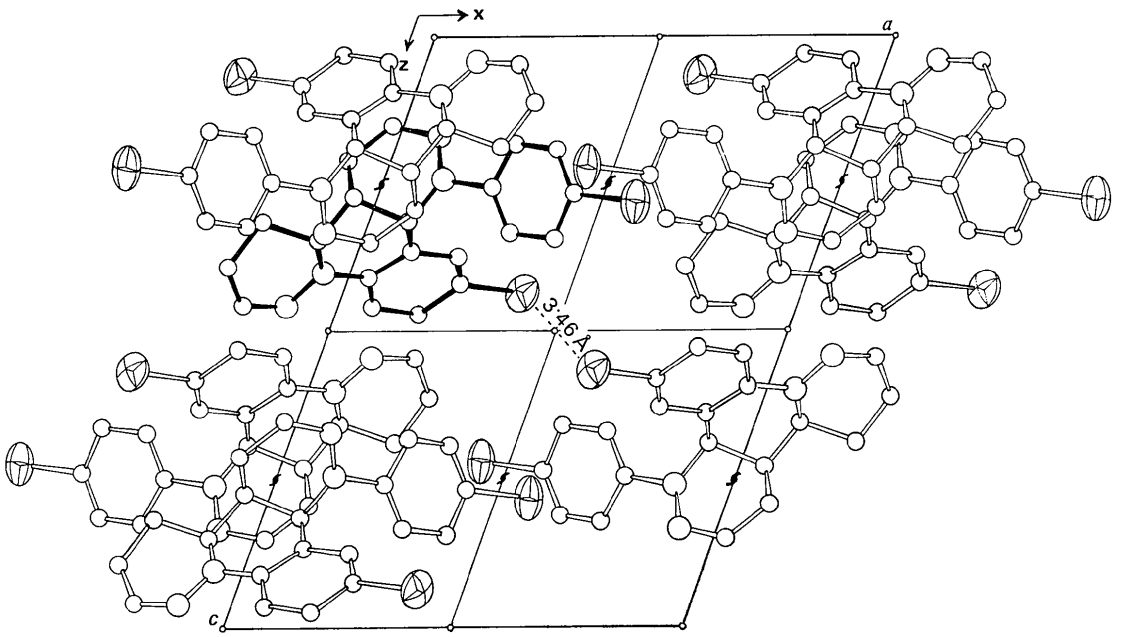

Fig. 5. The structure of HJBR-1 viewed along the *b* axis.

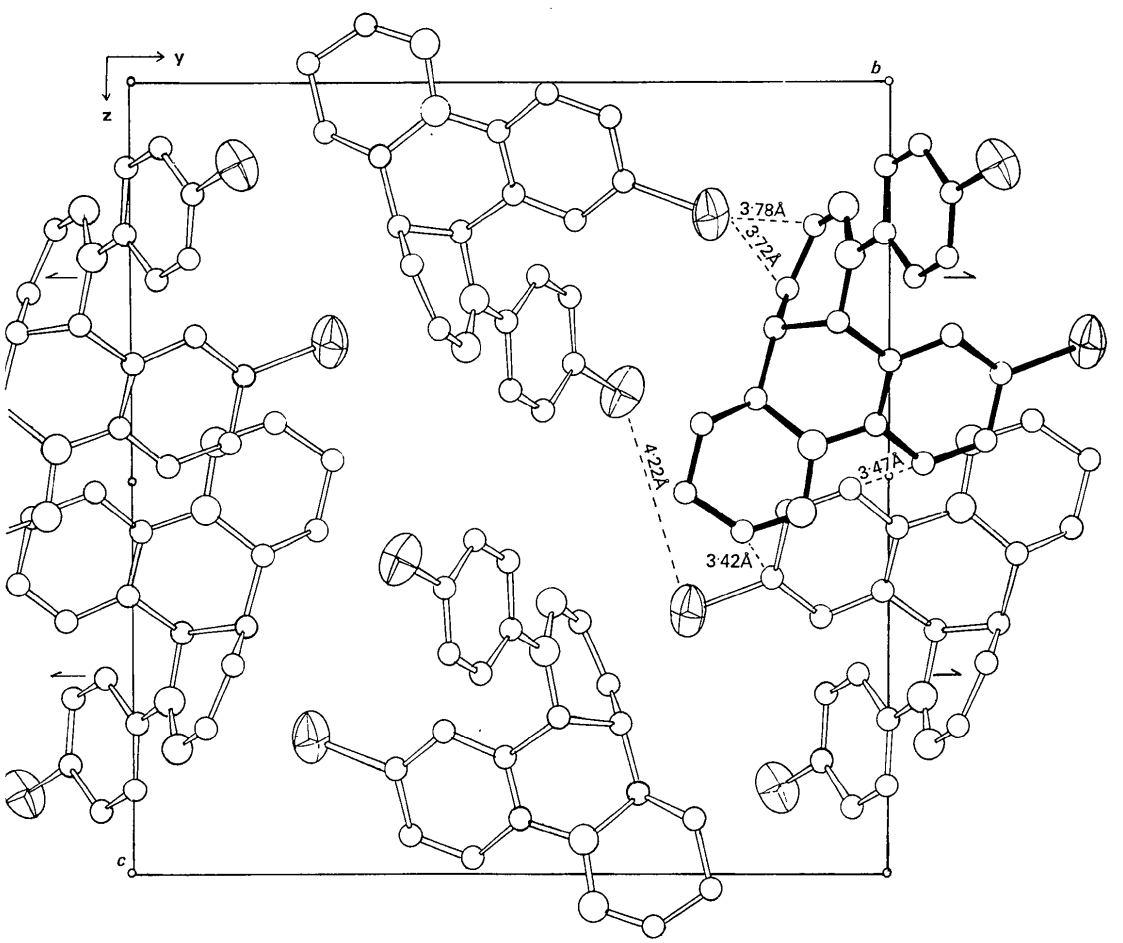

Fig. 6. The structure of HJBR-2 viewed along the *a** axis.

In addition the distance between Br(12) atoms, related by a centre of symmetry, is very short, 3·46 Å (*cf.* Fig. 5).

All the valency angles of HJBR-2 are quite normal. The opening of the angle C(13*b*)–N(1)–C(1′) (124°) may be ascribed to intramolecular steric strain between C(6′) and C(13). There are no short Br···Br distances in this structure (all of them are greater than 4·2 Å) and no Br···C or Br···N distances are less than 3·7 Å, and no C···N or C···C distances less than 3·4 Å (*cf.* Figs. 6 and 7).

In the structure of HJBR-1*a* all of the Br···Br distances are greater than 3·9 Å, but one rather short Br···N distance (3·43 Å) was found (*cf.* Fig. 8). In addition two short distances between light atoms were found. These are also indicated in Fig. 8.

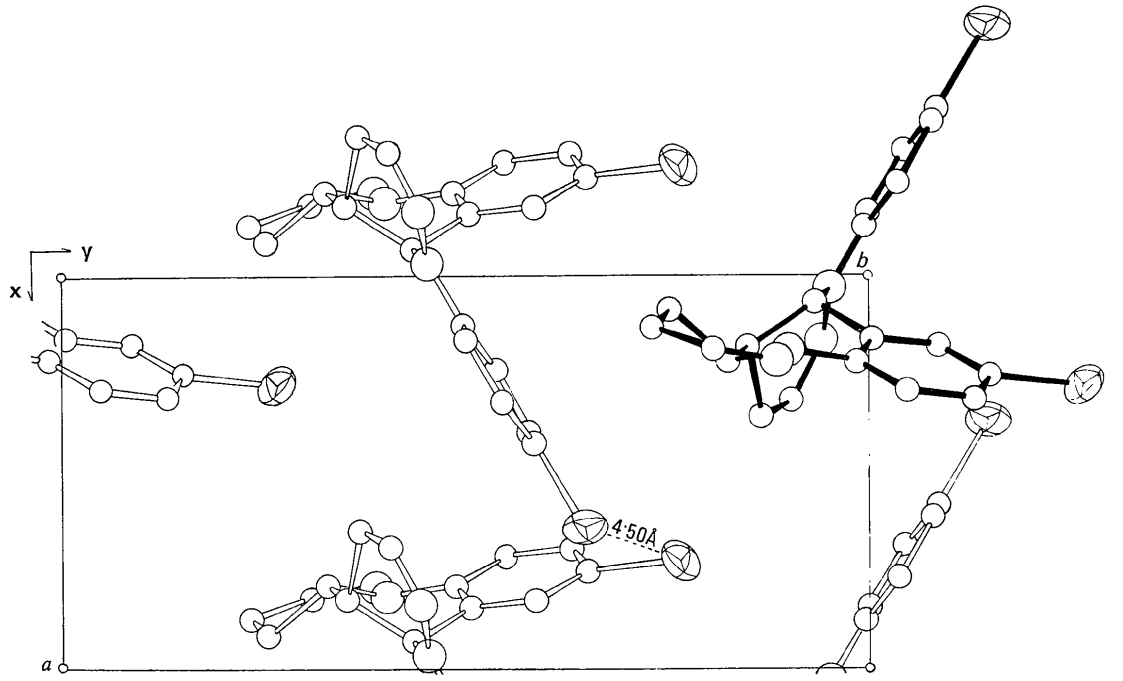

Fig. 7. The structure of HJBR-2 viewed along the *c* axis.

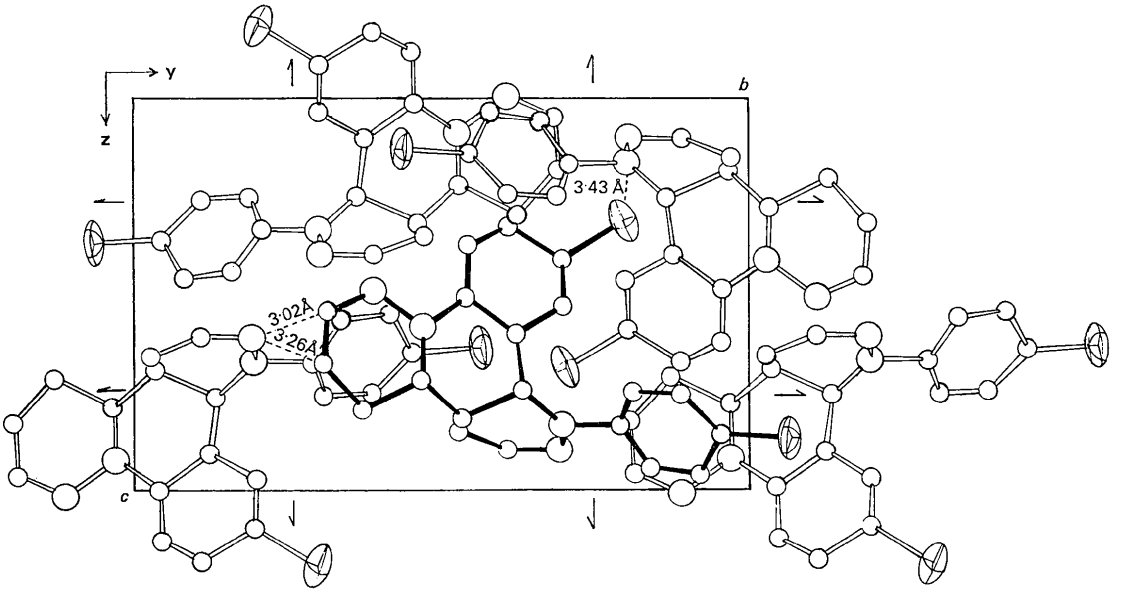

Fig. 8. The structure of HJBR-1*a* viewed along the *a* axis.

The molecules are packed in very different ways in the three crystals in spite of the very similar overall shape of the molecules. This can be seen by a comparison of Figs. 4 to 8. No close relation is found between the packing of the molecules in the racemate crystal, HJBR-1, and the packing in the corresponding chiral crystal HJBR-1*a*, as was the case in the structures reported by Cheng, Koo, Mellor, Nyburg & Young (1970). It seems likely that the packing of the HJBR-2 molecules is more favourable than the others. There are no short intermolecular distances in this structure, although the unit cell of HJBR-2 is the smallest one.

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The Crystal Structure of the *trans* Isomer of β-Ionylidenecrotonic Acid. II. Determination of Subsequent Data and Revaluation of Previous Results

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Following a previous paper on $C_{17}H_{24}O_2$, 9,10-*trans*-β-ionylidene-γ-crotonic acid, or conventionally *trans*-(2′,6′,6′-trimethylcyclohex-1′-enyl)-3-methylhexa-1,3,5-triene-6-carbooxylic acid, all crystal structure data have been determined with an automatic single-crystal diffractometer (Cu $K\alpha$ radiation) at room temperature. Space group $P\bar{1}$, $Z=2$. Cell constants: $a=10{\cdot}391$, $b=13{\cdot}481$, $c=7{\cdot}546$ Å, $\alpha=108{\cdot}12$, $\beta=127{\cdot}81$, $\gamma=68{\cdot}01°$. A least-squares anisotropic block-diagonal refinement was started from the previously published positional parameters of the carbon and oxygen atoms. Moreover all hydrogen atoms were refined, with individual isotropic B values. Final $R=0{\cdot}07$. The results allow a better comparison with those obtained more recently for the *cis* analogue and with details of other vitamin A and carotenoid related substances. The torsion angle between the ring-ethene system and the plane of the first three adjacent chain-carbon atoms is 10·4° from *s-trans*. Some possible physical interpretations of the very large anisotropic U_{ij} values of some ring atoms are discussed, in view of the significance of geometrical data in this and other related structures.

Introduction

This redetermination of the molecular and crystal structure and production of additional data of 9,10-*trans*-β-ionylidene-γ-crotonic acid, reported formerly in a paper by Eichhorn and MacGillavry (1959) has been undertaken in order to update the results. Comparison with the *cis* analogue (Eichhorn, 1957; Koch & MacGillavry, 1963; Koch, 1972) and with other vitamin A related (Stam & MacGillavry, 1963; Paul-Roy, Schenk & MacGillavry, 1969; Schenk,1969) and carotenoid related (Sly, 1964; Sterling, 1964; Bart & MacGillavry, 1968; Braun, Hornstra & Leenhouts, 1971) substances need an improved basis, in view of recent quantum, mechanical calculations (Pullman, Langlet & Berthod, 1969; Langlet, Pullman & Berthod, 1970) and semi-empirical calculations/nuclear magnetic resonance measurements (Honig, Hudson, Sykes & Karplus, 1971). Various experimental data on these compounds are also compared in the review articles by Hubbard & Wald (1968) and Schwieter, Englert, Rigassi & Vetter (1969).

The numbering of the carbon and oxygen atoms, used in this paper is given in Fig. 1 and that of the hydrogen atoms in Fig. 4.

Experimental

From a small single crystal (obtained from a 96% alcohol solution; m.p. 158 °C; dimensions 0·3 × 0·2 × 0·1