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Gaussian Profile Analysis in Computer Controlled Single Crystal Diffractometry

ROLF NORRESTAM

Institute of Inorganic and Physical Chemistry, University of Stockholm, S-104 05 Stockholm 50, Sweden

Single crystal X-ray reflections have been studied with an ω step-scan technique on a computer controlled linear diffractometer and intensities estimated by analysis of Gaussian profiles. Refinement of the crystal structure *versus* data so obtained yielded a conventional crystallographic R-value of 0.034.

The software of the diffractometer control system is described.

Linear single crystal diffractometers such as PAILRED [1] normally measure X-ray reflection intensities by scanning in ω (*i.e.* rotating the crystal with the counter stationary) and measure the background intensities at each end of the scan. Optimal use of this background-peak-background (BPB) method requires for each reflection that: I. The exact peak position is known. II. An estimate of the peak width is available to define the scan interval. III. Estimates of the total and background intensities are available to permit optimal division of the total time between counting times for total and background intensity measurements.[2]

Given accurate cell parameters and crystal orientation, one must either rely on high instrumental accuracy and on stability of the crystal orientation, or use a suitably enlarged scan interval to try to fulfil the first requirement. For, *i.a.*, PAILRED diffractometers, small backlashes in the angle settings cause minor deviations of the peak positions in ω.

Usually one tries to fulfil the second requirement by fitting a few premeasured peak-widths to some simple semi-empirical model. A preliminary determination of the intensities, to satisfy the third requirement, can also be used to decide which reflections should be considered as unobservable.[3]

The present paper describes an alternative procedure for use with a linear single-crystal diffractometer under closed-loop computer control.

DESCRIPTION OF SOME CHARACTERISTICS OF THE DIFFRACTOMETER SYSTEM USED

The present X-ray intensity measurements were made with a PAILRED linear single-crystal diffractometer under closed-loop control by an IBM 1800 computer operating under the IBM 1800 Time-Sharing Executive.[4] A detailed description of the control hardware and modifications developed in this institute for a commercially available PAILRED diffractometer has been given elsewhere.[5] The software for the diffractometer control, described briefly in a preliminary report,[6] is detailed below.

The diffractometer is controlled by signals sent *via* the digital output (DO) feature from the IBM 1800 computer.[7] Altogether 32 different DO-points, divided into two 16-bit words, are used, 20 for the output of a 5-digit binary-coded decimal (BCD) angle value, 4 to select different motor speeds, 5 to address the five different angle-setting motors (x, y, ω, μ, and ν), one to start and stop the scaler, and two to open and close the X-ray beam shutter.

Diffractometer input to the computer is *via* 11 digital input points treated as process interrupt (PI) contacts, 10 for input of the decimal digits (0 – 9) contained in each decade of the scaler, and one to tell the computer whether all motors have stopped or not.

The setting of an angle is performed by digital output of the desired 5-digit BCD angle value together with two more signals to identify motor and

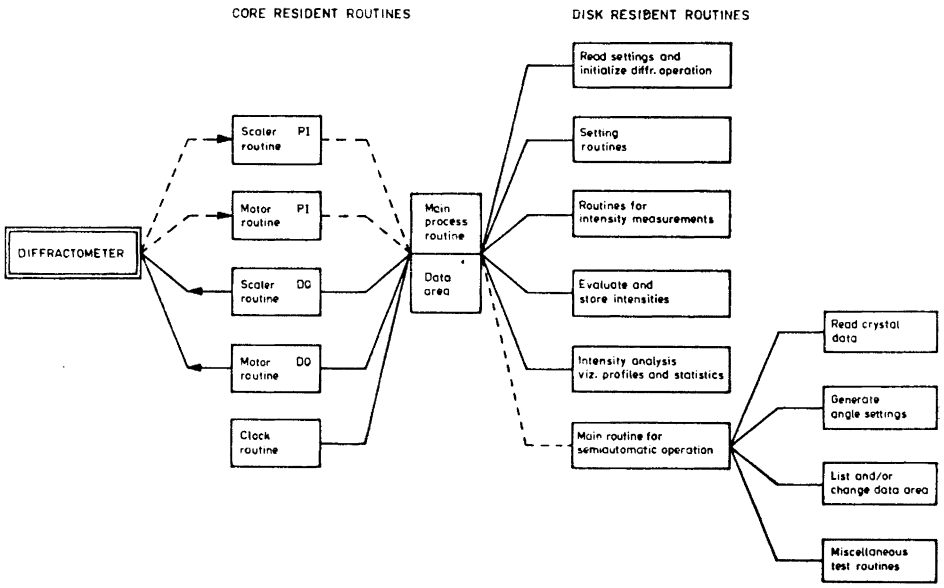

Fig. 1. Schematic drawing of the linkages among the routines of the program system for diffractometer control. The dashed lines indicate that the corresponding links are initiated by transmission of process interrupts rather than by direct calls.

speed. The desired angle value is compared electronically, by circuits in the diffractometer interface, with the current value of the angle setting. The current setting is changed towards the desired value; when coincidence occurs the angle-setting is stopped and a process interrupt informs the computer that the setting is completed.

As mentioned, the scaler is activated by one signal from the computer. When a "stop scaler" signal is received by the diffractometer, the 7 decades of the scaler give rise to 7 consecutive process interrupts at the computer. These 7 interrupts transmit the 7 digit decimal scaler content.

The diffractometer input/output routines, written in assembler language, reside permanently in core memory to permit efficient data flow. A small data area (presently 100 16-bit words) also resides permanently in core memory to permit fast communication between different programs of the system.

Fig. 1 shows the linking between the routines of the diffractometer system. Entry of the PI routines is triggered by signals from the diffractometer. The PI routines use the computer's programmed interrupt feature to call the main process routine. The action of this main routine is determined by the current contents of the data area. This area thus forms a link between all the programs of the system. The main routine for semiautomatic operation is entered by a console interrupt at the computer. By giving suitable programmed interrupts, this main routine calls in several input/output routines (*e.g.* read crystal data from cards, generate angles settings, list and/or change the content of the data area in core) and routines to test specific parts of the diffractometer control system.

PROCEDURE FOR EXAMINING REFLECTIONS

A diffractometer under closed-loop control can make scan of a reflection to yield parameters for measuring the integrated net intensity by the BPB-method with the best division of the total time. Estimates of the peak-width and the reflection centroid position (in the scanned angle) can also be obtained if the intensity scan is performed stepwise. The preliminary scan interval should be large enough so that all the reflection profile is covered. The design of the PAILRED instrument used in the present study permits peak positional changes to be expressed only in terms of ω during on-line control of the diffractometer. A preliminary step scan in ω over the reflection thus gives all the information needed to permit the choice of a good measuring strategy, provided that some physically reasonable model of the reflection profile is used.

Several theoretical and sophisticated empirical models, have been suggested (see, *e.g.*, Wilson [8] and Diamond [9]) for determination of integrated net intensities. Since our original intention in analyzing the step-scan measurements was simply to choose measuring strategy and not to determine intensities, simple Gaussian reflection profiles were used. Gaussian profiles are physically reasonable if the scan is performed in ω and not in the direction of the spectral streak, *e.g.* $\theta - 2\theta$, and have recently been successfully applied to empirical analysis of powder diffraction data.[10]

A Gaussian reflection profile can be written:

$$I(\omega) = C_1 \exp\left[-4 \ln 2 \left(\frac{\omega - \omega_0}{C_2}\right)^2\right] \quad (1)$$

where C_1 is the maximum peak height at the centroid position ω_0, and where C_2 is the full width at half the maximum peak height. If the logarithm of (1) is taken, one obtains

$$\ln [I(\omega)] = -\frac{4 \ln 2}{C_2^2} (\omega - \omega_0)^2 + \ln [C_1] \quad (2)$$

so that $\ln [I(\omega)]$ is a linear function of $(\omega - \omega_0)^2$, $\ln [I(\omega)] = a(\omega - \omega_0)^2 + b$, with slope $a = -(4 \ln 2)/C_2^2$ and intercept $b = \ln [C_1]$. Thus, $C_2 = [-(4 \ln 2)/a]^{\frac{1}{2}}$ and $C_1 = \exp [b]$. Given the e.s.d.'s $\sigma(a)$ and $\sigma(b)$ for a and b, respectively, the e.s.d.'s of C_1 and C_2 can be estimated as

$$\sigma(C_1) = \exp [b] \times \sigma(b) \text{ and } \sigma(C_2) = \frac{C_2}{2a} \times \sigma(a) \quad (3)$$

Since $\int_{-\infty}^{\infty} \exp [-at^2] dt = (\pi/a)^{\frac{1}{2}}$ it follows that

$$I \equiv \int_{-\infty}^{\infty} I(\omega) d(\omega - \omega_0) = C_1 \ C_2 \left(\frac{\pi}{4 \ln 2}\right)^{\frac{1}{2}} \quad (4)$$

and the e.s.d. of I can be estimated as

$$\sigma(I) = \left\{\frac{\pi}{4 \ln 2} [C_1^2 \ \sigma^2(C_1) + C_2^2 \ \sigma^2(C_2)]\right\}^{\frac{1}{2}} \quad (5)$$

In the present study the value of ω_0 has been calculated as

$$\omega_0 = \int_{\omega_1}^{\omega_2} \omega I(\omega) \ d\omega / \int_{\omega_1}^{\omega_2} I(\omega) d\omega \quad (6)$$

i.e. the center of mass of $I(\omega)$ in the range $\omega_1 \leq \omega \leq \omega_2$, by substituting the corresponding sums for the integrals in (6), calculated from the net intensities obtained at the discrete ω-values used in the step-scan interval.

The net intensities $I(\omega_i)$ at the n different ω-values in the scan interval, used in formulas (6) and (2), have been calculated from the corresponding total intensities N_i as

$$I(\omega_i) = N_i - \min(N_1, N_2, \ldots, N_n) \quad (8)$$

This might appear an oversimplification. However, fitting the obtained estimated net intensities to a predicted physically reasonable model decreases the errors introduced, if a proper weighting of the observations is applied.

The estimated intensities and the centroid ω_0 from formulas (7) and (6) have been fitted to the linear expression (2) by the method of least-squares. Of several different weighting formulas studied, it was found that weights (W_i)

$$W_i = N_i^2 \tag{8}$$

(where N_i are the measured total intensities) gave acceptable results both for strong and weak reflections. This weighting was thus adopted, and its applicability was fully supported by the outcome of the investigation.

APPLICATION OF GAUSSIAN PROFILE

The procedures outlined above for determination of X-ray reflection characteristics were applied to a crystal of 4a-allyl-3,5,7,8,10-pentamethyl-4a,5-dihydroisoalloxazine [11] ($C_{18}H_{22}N_4O_2$). The unit cell of this compound is orthorhombic, space group $P2_12_12_1$ ($Z=4$) with the dimensions $a=17.422(5)$, $b=12.777(3)$, and $c=7.564(5)$Å. The crystal selected, of dimensions $0.20\times0.30\times0.22$ mm^3 with the prismatic axis along the b-direction, was mounted on a goniometer along this axis. A total of 704 reflections (**k** ranging from 0 to 9) were collected on the PAILRED diffractometer with graphite monochromatized Cu$K\alpha$ radiation ($\lambda=1.54184$ Å) and a scintillation detector with pulse height discrimination. The ω scan range used was 2.4°, covered by nine steps of 0.3° each, 10 seconds measurement per step. Since the reflection-widths ranged from 1.2 to 1.4°, only $4-5$ of the 9 measured points in the step-scan were in the significant reflection range. The large scan interval (2.4°) was selected to assure that the reflections would lie within the scan-interval throughout the whole investigation, without any adjustments of either the crystal or the instrument. The fairly long measuring times (10 s) at each step and the small number of steps (9), were chosen as a compromise between obtaining bad counting statistics for a large number of steps and good statistics for a small number of steps, within a reasonable time, 90 s. The choice of the fixed measuring time of 10 s per step was also dependent on the transmission time of the scaler contents, about 1 s, inherent in the hardware.

Fig. 2 illustrates the computer output for the reflection (2 8 2). The centroid position ω_0 is -11.6×10^{-2}, ($\omega=0$ corresponds to the expected position). The least-squares (LS) fit to the linearized expression (2) gives $a=-0.00054(3)$ and $b=4.30(3)$, with a correlation coefficient for the LS line of -0.988. The constants C_1 and C_2 in formula (2) were calculated to be 74(2) and 72(2), respectively. In the evaluation of the integrated intensity and its e.s.d., the proportionality constant 0.1 instead of $[\pi/(4\ln 2)]$ was used in formulas (4) and (5). As seen from Fig. 2, and also indicated by the high correlation factor, the agreement between the net intensities, observed and calculated per (1), is encouraging. Note that the ω values given are multiplied by 10^2, while the net intensities have been normalized to give the number of counts per second.

The promising outcome of the use of Gaussian function for profile analysis of the reflections led us to collect 709 intensities from the crystal, evaluated from profiles rather than from the BPB-measurements originally intended. The data were corrected for Lp and absorption effects ($\mu=7.1$ cm^{-1}). After five cycles of least-squares refinement of one scale factor and the positional

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*REFLECTION NR.*  675
*INDICES*   2   8   2
*CENTROID*   -11.6

*LS-LINE* -0.000540 ( 0.000031 )          4.299 (  0.025 )          -0.988
*GAUSS-PROFILE*   71.63 (    2.06 )           73.6 (     1.8 )
*GAUSS-INTENSITY*            52.7 (     2.0 )    SIGMA(I)/I  0.038
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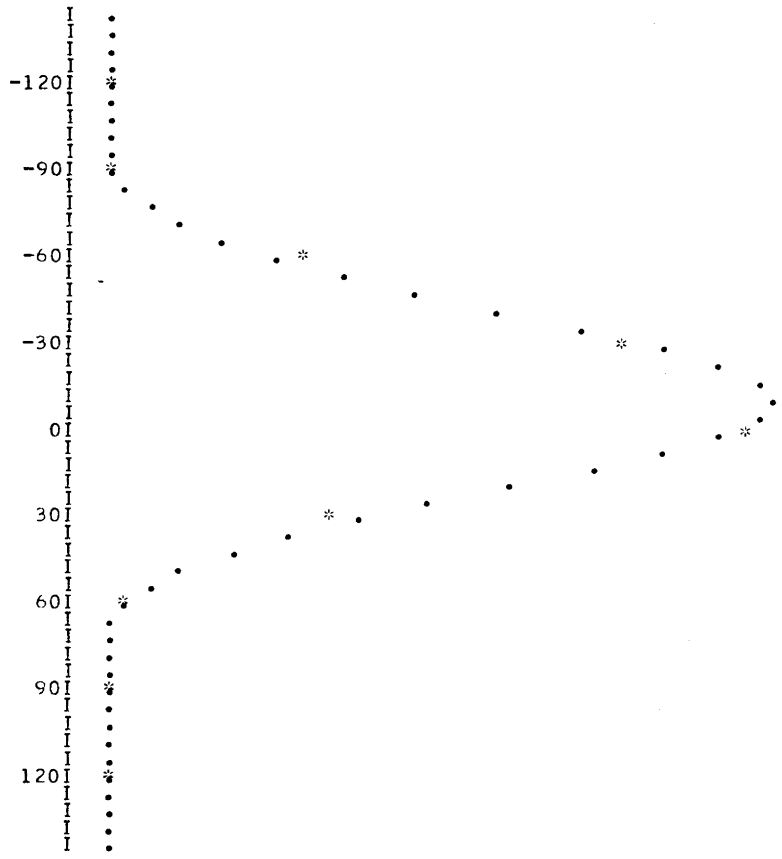

OMEGA.	I(NET)OBS.	I(NET)CALC.	DELTA.
-120	0.0	0.1	-0.1
-90	2.0	2.6	-0.6
-60	23.0	20.7	2.2
-30	58.0	61.3	-3.3
0	71.0	68.4	2.5
30	27.0	28.8	-1.8
60	4.0	4.6	-0.6
90	1.0	0.2	0.7
120	0.0	0.0	0.0

Fig. 2. Computer output for the reflection with indices (2 8 2).

Table 1. Comparison between observed and calculated structure factors for 4a-allyl-3,7,8,10-pentamethyl-4a,5-dihydroisoalloxazine. The observed structure factors were derived from Gaussian profile analysis of reflections measured by a computer controlled PAILRED diffractometer with monochromatized Cu$K\alpha$ radiation (X-ray collimator diameter: 1.5 mm, detector aperture width 3°). Each group of three columns contains k, $10|F_o|$ and $10|F_c|$, and is headed by the values of h and k common to the group.

H= 15 K= 0		
1	321	312
H= 14 K= 0		
2	74	73
1	174	172
0	447	464
H= 13 K= 0		
1	48	49
2	174	174
3	64	65
H= 12 K= 0		
4	48	46
3	25	23
2	94	96
1	122	121
0	329	345
H= 11 K= 0		
1	42	42
2	177	177
3	38	40
4	23	21
H= 10 K= 0		
5	123	121
4	134	129
3	164	160
2	136	140
1	159	151
0	134	139
H= 9 K= 0		
1	239	252
2	88	80
3	256	260
4	118	114
5	22	18
H= 8 K= 0		
5	0	5
4	200	199
3	213	206
2	261	248
1	127	115
0	88	87
H= 7 K= 0		
1	207	189
2	212	199
3	132	132
4	95	92
5	214	208
H= 6 K= 0		
6	41	42
5	120	114
4	45	42
3	159	154
2	185	189
1	115	113
0	202	189
H= 5 K= 0		
1	262	231
2	253	261
3	88	84
4	57	55
5	97	93
6	93	95
H= 4 K= 0		
6	85	83
5	90	93
4	48	46
3	155	153
2	34	33
1	72	68
H= 3 K= 0		
1	495	448
2	160	164
3	244	238
4	56	57
5	162	159
6	96	90
H= 2 K= 0		
6	55	53
5	67	65
4	31	30
3	288	262
2	454	458
1	319	332
H= 1 K= 0		
2	499	540
3	195	193
4	266	264
5	177	182
6	102	99
H= 0 K= 0		
6	369	373
4	327	336
2	461	483
H= 15 K= 1		
0	327	328
1	452	451
H= 14 K= 1		
2	327	323
1	155	162
0	421	426
H= 13 K= 1		
0	60	58
1	42	46
2	262	258
3	39	38
H= 12 K= 1		
4	34	35
3	97	97
2	164	174
1	127	136
0	175	192
H= 11 K= 1		
0	18	17
1	188	199
2	44	42
3	147	142
4	23	22
H= 10 K= 1		
4	56	55
3	146	148
2	18	19
1	142	139
0	82	83
H= 9 K= 1		
0	109	113
1	74	73
2	206	206
3	133	132
4	37	36
5	111	107
H= 8 K= 1		
5	92	89
4	252	247
3	61	56
2	82	87
1	147	138
0	303	305
H= 7 K= 1		
0	46	47
1	621	567
2	360	338
3	385	379
4	157	156
5	142	140
H= 6 K= 1		
6	191	195
5	83	82
4	159	160
3	174	178
2	122	122
1	291	263
0	196	189
H= 5 K= 1		
0	338	320
2	273	273
3	157	147
4	81	80
5	167	156
6	116	116
H= 4 K= 1		
6	93	90
5	63	62
4	103	106
3	200	202
2	205	210
1	207	175
0	97	98
H= 3 K= 1		
0	25	24
1	245	234
2	513	554
3	214	221
4	87	84
5	88	77
6	36	36
H= 2 K= 1		
6	107	110
5	84	84
4	214	208
3	90	91
2	435	438
1	220	218
0	286	311
H= 1 K= 1		
2	635	683
3	116	118
4	161	162
5	156	152
6	71	71
H= 0 K= 1		
6	186	184
5	169	163
4	205	206
3	252	251
1	64	64
H= 15 K= 2		
0	140	142
H= 14 K= 2		
2	257	267
1	327	337
0	225	232
H= 13 K= 2		
0	50	54
1	90	85
2	102	104
3	131	130
H= 12 K= 2		
3	77	76
2	87	89
1	98	98
0	39	35
H= 11 K= 2		
0	175	171
1	23	22
2	131	129
3	102	103
4	10	9
H= 10 K= 2		
4	116	121
3	151	147
2	123	128
1	210	212
0	154	158
H= 9 K= 2		
0	232	241
1	60	58
2	69	66
3	79	78
4	26	24
5	103	108
H= 8 K= 2		
5	61	60
4	143	141
3	23	22
2	343	337
1	377	343
0	205	209
H= 7 K= 2		
0	527	526
1	436	421
2	102	94
3	311	310
4	91	93
5	132	129
H= 6 K= 2		
5	43	40
4	152	152
3	95	95
2	183	182
1	249	231
0	302	295
H= 5 K= 2		
0	38	35
1	305	281
2	284	290
3	182	191
4	146	148
5	116	121
6	30	28
H= 4 K= 2		
6	73	73
5	96	94
4	146	144
3	58	57
2	332	346
1	382	347
0	197	195
H= 3 K= 2		
0	37	4
1	282	273
2	189	193
3	88	87
4	117	114
5	74	75
6	18	19
H= 2 K= 2		
6	88	90
5	118	127
4	161	162
3	288	291
2	316	306
1	460	462
0	63	62
H= 1 K= 2		
1	313	322
2	618	674
3	522	551
4	202	205
5	39	39
6	76	72
H= 0 K= 2		
6	148	148
5	192	190
4	461	470
3	221	223
H= 14 K= 3		
2	228	235
1	208	221
0	124	119
H= 13 K= 3		
0	19	19
1	97	100
2	142	142
3	107	110
H= 12 K= 3		
3	0	12
2	39	40
1	80	81
0	0	5
H= 11 K= 3		
0	63	66
1	25	24
2	35	34
3	69	67
4	126	125
H= 10 K= 3		
4	57	59
3	57	58
2	39	40
1	76	70
0	8	6
H= 9 K= 3		
0	106	107
1	184	188
2	55	56
3	84	78
4	114	119
5	34	32
H= 8 K= 3		
5	114	116
4	115	113
3	267	270
2	291	274
1	114	104
0	416	419
H= 7 K= 3		
0	137	137
1	582	575
2	43	40
3	56	55
4	213	215
5	33	27
H= 6 K= 3		
5	62	62
4	33	31
3	179	173
2	152	145
1	142	136
0	54	54
H= 5 K= 3		
0	165	162
1	272	263
2	192	185
3	179	174
4	127	131
5	87	88
6	116	114
H= 4 K= 3		
6	56	56
5	41	41
4	174	159
3	154	157
2	79	81
1	346	327
0	159	153
H= 3 K= 3		
0	287	298
1	320	325
2	289	310
3	125	125
4	48	51
5	82	77
6	61	61
H= 2 K= 3		
6	29	25
5	81	80
4	238	247
3	253	242
2	406	420
0	36	33
H= 1 K= 3		
2	497	537
3	310	324
4	297	302
5	286	295
6	205	208
H= 0 K= 3		
6	67	65
5	323	314
4	530	535
3	239	240
2	434	455
1	145	138
H= 14 K= 4		
1	54	54
0	83	80
H= 13 K= 4		
0	0	6
1	134	132
2	67	63
H= 12 K= 4		
3	132	127
2	28	26
1	58	61
0	94	91
H= 11 K= 4		
0	38	37
1	82	79
2	89	88
3	39	38
4	18	16
H= 10 K= 4		
4	85	87
3	43	41
2	35	31
1	100	90
0	124	125
H= 9 K= 4		
0	101	103
1	108	96
2	183	181
3	138	137
4	62	63
H= 8 K= 4		
5	62	61
4	74	70
3	140	142
2	118	113
1	117	117
0	660	652
H= 7 K= 4		
0	377	374
1	717	715
2	147	137
3	144	156
4	107	106
5	217	215
H= 6 K= 4		
5	80	79
4	43	36
3	155	155
2	109	107
1	334	303
0	269	257
H= 5 K= 4		
0	156	157
1	124	114
2	316	308
3	49	50
4	25	21
5	105	103
H= 4 K= 4		
5	38	37
4	71	72
3	246	243
2	51	48
1	249	244
0	114	110
H= 3 K= 4		
0	242	243
1	175	172
2	127	127
3	120	116
4	151	151
5	68	66
6	109	109
H= 2 K= 4		
6	92	97
5	86	88
4	298	315
3	227	227
2	187	173
1	341	354
H= 1 K= 4		
1	361	358
2	480	503
3	446	447
4	292	296
5	194	192
6	101	102
H= 0 K= 4		
6	170	166
5	66	66
4	212	214
2	383	383
1	73	73
H= 13 K= 5		
0	95	93
1	123	122
2	65	63
H= 12 K= 5		
3	26	27
2	88	91
1	143	140
0	53	49
H= 11 K= 5		
0	54	52
1	34	38
2	128	131
3	63	63
H= 10 K= 5		
4	80	80
3	32	30
2	65	63
1	55	56
0	23	19
H= 9 K= 5		
0	84	85
1	96	102
2	146	139
3	64	60
4	103	101
H= 8 K= 5		
4	208	209
3	143	142
2	254	243
1	232	219
0	101	96
H= 7 K= 5		
0	49	49
1	393	385
2	115	117
3	54	56
4	63	65
5	234	233
H= 6 K= 5		
5	70	71
4	67	67
3	101	99
2	165	169
1	174	166
0	304	287
H= 5 K= 5		
0	97	98
1	184	177
2	144	143
3	97	98
4	148	146
5	91	85
H= 4 K= 5		
5	82	82
4	71	66
3	112	118
2	49	45
1	145	142
0	59	56
H= 3 K= 5		
0	295	288
1	203	194
2	45	43
3	59	57
4	35	32
5	110	104
H= 2 K= 5		
5	30	28
4	170	172
3	115	118
2	169	153
1	164	154
0	173	175
H= 1 K= 5		
1	295	295
2	110	109
3	316	325
4	76	74
5	151	162
6	32	35
H= 0 K= 5		
6	151	162
5	118	119
4	162	162
3	239	250
2	523	509
1	426	437
H= 13 K= 6		
1	102	98
0	15	12
H= 12 K= 6		
0	120	121
1	81	81
2	66	67
H= 11 K= 6		
3	146	140
2	33	28
1	39	35
0	128	125
H= 10 K= 6		
0	9	7
1	108	110
2	56	53
3	81	80
H= 9 K= 6		
4	78	79
3	176	176
2	112	109
1	41	40
0	45	44
H= 8 K= 6		
0	127	127
1	203	200
2	339	336
3	78	78
4	186	186
H= 7 K= 6		
4	371	373
3	381	372
2	218	204
1	81	79
0	290	282
H= 6 K= 6		
0	42	40
1	173	164
2	37	35
3	166	162
4	106	102
5	159	164
H= 5 K= 6		
5	104	103
4	121	118
3	80	87
2	39	40
1	74	76
0	130	133
H= 4 K= 6		
0	119	122
1	112	105
2	55	56
3	95	92
4	194	185
5	40	42
H= 3 K= 6		
5	58	56
4	198	201
3	137	125
2	74	72
1	52	52
0	26	27
H= 2 K= 6		
0	122	121
1	201	189
2	82	79
3	67	60
4	107	103
5	137	143
H= 1 K= 6		
5	136	131
4	177	192
3	82	84
2	243	229
1	44	41
H= 0 K= 6		
1	473	504
2	172	164
3	244	242
4	37	35
5	0	3
H= 12 K= 7		
0	59	61
1	53	55
H= 11 K= 7		
2	26	27
1	79	78
0	8	10
H= 10 K= 7		
0	29	27
1	35	29
2	110	110
3	35	32
H= 9 K= 7		
3	126	129
2	174	177
1	89	88
0	13	12
H= 8 K= 7		
0	13	3
1	54	50
2	207	201
3	297	303
4	216	203
H= 7 K= 7		
4	298	298
3	75	78
2	135	132
1	177	172
0	0	7
H= 6 K= 7		
0	145	143
1	46	47
2	242	240
3	59	56
4	176	169
H= 5 K= 7		
4	65	64
3	123	127
2	73	73
1	144	144
0	52	50
H= 4 K= 7		
0	44	45
1	73	71
2	237	220
3	55	53
4	93	97
5	62	61
H= 3 K= 7		
5	79	80
4	31	31
3	47	44
2	21	21
1	91	85
0	71	68
H= 2 K= 7		
0	84	87
1	213	202
2	171	156
3	23	24
4	165	164
5	87	86
H= 1 K= 7		
5	40	36
4	44	46
3	256	249
2	348	326
1	111	107
H= 0 K= 7		
1	407	383
2	206	202
3	45	42
4	319	313
5	163	154
H= 11 K= 8		
0	48	46
1	70	69
H= 10 K= 8		
2	58	55
1	40	39
0	71	72
H= 9 K= 8		
0	95	94
1	99	97
2	102	104
3	45	45
H= 8 K= 8		
3	198	194
2	94	91
1	153	146
0	306	304
H= 7 K= 8		
0	37	33
1	153	149
2	324	316
3	418	416
4	117	107
H= 6 K= 8		
4	265	268
3	129	131
2	201	200
1	84	79
0	34	34
H= 5 K= 8		
0	60	60
1	155	151
2	61	61
3	7	7
4	101	108
H= 4 K= 8		
4	147	153
3	92	91
2	76	75
1	77	80
0	41	41
H= 3 K= 8		
0	6	6
1	41	40
2	103	96
3	51	49
4	53	55
H= 2 K= 8		
5	73	75
4	69	69
3	34	31
2	64	63
1	186	184
0	189	197
H= 1 K= 8		
1	73	70
2	160	155
3	27	26
4	157	154
5	123	128
H= 0 K= 8		
5	13	0
4	80	81
3	184	188
H= 10 K= 9		
1	28	25
0	0	10
H= 9 K= 9		
0	27	26
1	175	187
2	14	11
H= 8 K= 9		
3	76	75
2	307	316
1	127	134
0	183	190
H= 7 K= 9		
0	81	84
1	260	265
2	175	185
3	196	205
H= 6 K= 9		
3	142	139
2	193	195
1	146	154
0	238	257
H= 5 K= 9		
0	126	125
1	48	46
2	151	161
3	110	111
4	22	23
H= 4 K= 9		
4	44	47
3	62	65
2	58	60
1	64	68
0	124	131
H= 3 K= 9		
0	5	5
1	44	44
2	97	101
3	79	77
4	58	61
H= 2 K= 9		
4	18	18
3	136	132
2	112	107
1	38	37
H= 1 K= 9		
2	91	85
3	31	31
4	67	66
H= 0 K= 9		
4	97	100
3	113	112

and thermal parameters of the nonhydrogen atoms (217 refined parameters altogether) the conventional crystallographic R-value ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$) fell to 0.034. In this refinement the positional and thermal parameters of the 22 hydrogens were kept fixed at the values determined earlier.[11] The five strongest reflections, measured with counting rates above 10 000 counts/s, were omitted because of coincidence losses in the detector system. The observed and calculated structure factors are listed in Table 1. The resultant thermal and positional parameters agree within a few e.s.d.'s with those obtained [11] from refinement *versus* 1606 reflections measured by $\theta - 2\theta$ scans (BPB method) with a Siemens AED single crystal diffractometer. Similar refinement *versus* the structure factors of the same 704 reflections measured with the Siemens diffractometer yielded an R-value of 0.028. The somewhat lower R-value obtained in the latter case is reasonable since each reflection was here measured for up to 768 s, compared to the 90 s on the PAILRED instrument.

In Table 2 the R values for different ranges of structure factor magnitudes, $|F|$'s, and for the 10 different layers ($k = 0 - 9$) are given for the two data sets. The very small $|F|$'s show poorer agreement between observed and calculated values in both methods of measurements. The absolute differences in R values are obviously attributable to the large difference in measuring time between the two data sets, rather than to the difference in measuring methods (profile

Table 2. Distribution of linear crystallographic R values *versus* structure factor magnitudes $|F|$ (40 – 80 reflections per entry), and for the 10 different layers, $h0l$ to $h9l$. The columns designated PAILRED represent the data obtained by Gaussian profile analysis of measurements on the linear PAILRED diffractometer, while those designated SIEMENS represent the data obtained by BPB measurements on the Siemens three-circle diffractometer.

$\|F\|$ range	PAILRED	SIEMENS
0 – 4	0.082	0.063
4 – 6	0.037	0.031
6 – 8	0.023	0.023
8 – 10	0.030	0.026
10 – 12	0.031	0.029
12 – 14	0.026	0.027
14 – 16	0.029	0.027
16 – 20	0.031	0.029
20 – 24	0.034	0.019
24 – 30	0.033	0.028
> 30	0.035	0.027
Layer	**PAILRED**	**SIEMENS**
0	0.039	0.030
1	0.035	0.030
2	0.034	0.023
3	0.035	0.033
4	0.026	0.029
5	0.031	0.026
6	0.033	0.030
7	0.036	0.024
8	0.026	0.024
9	0.043	0.023

analysis *versus* BPB-method) or in diffractometer geometry (equi-inclination *versus* normal-beam equatorial geometry). The R values for the 10 different layers of the PAILRED data do not show any remarkable systematic variations. Thus, the distribution of the R values for the different $|F|$ ranges and for different equi-inclination angles (μ) of the PAILRED data, obtained by Gaussian profile analysis, indicate no serious systematic variation of the errors, neither with the magnitude of $|F|$ nor with the value of μ.

The possible complications when applying Gaussian profiles to measurements with Mo$K\alpha$ radiation, due to the larger $\alpha_1 - \alpha_2$ splitting in Mo$K\alpha$ radiation compared with Cu$K\alpha$, are currently under investigation.

CONCLUSIONS

The fitting described above of a few ω step-scan intensity measurements of a reflection to a Gaussian profile sufficed to yield net intensities of such quality that they could be used for accurate determination of the crystal structure. Profile analysis has the advantage of readily permitting checking of error conditions during on-line measurements. For example, examination of the peak position provides prompt indication of crystal movements and miss-settings of the instrument, while the correlation between observed net intensities and intensities calculated from the determined profile-parameters (by formula (1)) can be used to assess whether the reflections have physically reasonable profiles. If higher accuracy is desired, one could either use a fast preliminary profile analysis for each reflection to obtain the parameters needed for optimal BPB measurements or, alternatively, increase the number of steps in the step scan and/or optimize the time spent on each step to obtain a better determination of the profile.

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