Table 2. *Observed and calculated* $10^5.\sin^2\theta$ *values of* $M_2Co(SO_4)_2.6H_2O$

	M = K			M = Rb			M = NH_4			M = Cs	
	$10^5.\sin^2\theta$			$10^5.\sin^2\theta$			$10^5.\sin^2\theta$			$10^5.\sin^2\theta$	
hkl	obs.	calc.	*hkl*	obs.	calc.	*hkl*	obs.	calc.	*hkl*	obs.	calc.
020	1601	1592	110	1144	1146	020	1518	1514	110	1110	1109
001	1694	1677	020	1544	1535	001	1670	1667	001	1602	1603
011	2074	2075	001	1661	1655	011	2048	2046	011	1963	1964
11$\bar{1}$	2266	2267	11$\bar{1}$	2189	2181	11$\bar{1}$	2148	2146	120	2192	2192
200	3096	3092	120	2300	2297	120	2276	2273	021	3048	3046
021	3271	3270	200	3046	3048	200	3033	3036	12$\bar{1}$	3154	3149
111	3420	3430	021	3192	3190	021	3182	3181	20$\bar{1}$	3305	3307
20$\bar{1}$	3598	3606	210	3435	3432	12$\bar{1}$	3282	3281	210	3363	3355
21$\bar{1}$	4009	4004	130	4209	4216	20$\bar{1}$	3378	3385	111		3358
130	4348	4356	220	4580	4583	111	3470	3464	21$\bar{1}$	3667	3667
121	4624	4625	13$\bar{1}$	5251	5251	21$\bar{1}$	3766	3764	130	3997	3995
031	5261	5260	201	5935	5941	130	4170	4166	220	4435	4437
13$\bar{1}$	5450	5451	040	6145	6140	220	4558	4550	121		4440
201	5937	5933	211	6325	6325	121	4600	4599	13$\bar{1}$	4954	4953
211	6350	6331	230	6508	6502	22$\bar{1}$	4895	4899	040	5771	5772
040		6370	140	6904	6902	031	5078	5074	201	5891	5888
002	6711	6711	31$\bar{1}$	7030	7038	13$\bar{1}$	5174	5174	11$\bar{2}$	6233	6232
11$\bar{2}$		6717	20$\bar{2}$	7184	7188	201	6020	6021	31$\bar{1}$	6771	6764
310	7367	7355	310	7243	7241	040	6061	6056	012		6774
221	7511	7525	221	7466	7476	211	6399	6400	20$\bar{2}$	6821	6826
21$\bar{2}$	7894	7872	21$\bar{2}$	7572	7572	11$\bar{2}$	6491	6488	310	7097	7097
12$\bar{2}$		7911	12$\bar{2}$	7681	7677	131		6492	12$\bar{2}$	7322	7314
14$\bar{1}$	8236	8238	041	7801	7795	140	6815	6815	221		7331
32$\bar{1}$	8487	8481	14$\bar{1}$	7929	7938	31$\bar{1}$	6899	6900	141	7485	7479
231	9521	9516	32$\bar{1}$	8183	8189	20$\bar{2}$	7068	7069	32$\bar{1}$	7837	7847
33$\bar{1}$	10468	10472	320	8397	8393	310	7211	7210	320	8181	8180
241	12299	12303	22$\bar{2}$	8712	8723	221	7535	7537	240	8771	8766
250	13060	13044	240	9188	9188	12$\bar{2}$	7621	7624	141		8770
042		13079	231	9388	9395	32$\bar{1}$	8033	8034	24$\bar{1}$	9103	9079
24$\bar{2}$	13840	13844	13$\bar{2}$	9601	9595	112	9120	9124	13$\bar{2}$		9118
420	13963	13960	24$\bar{1}$		9604	231	9429	9428	31$\bar{2}$	9462	9638
331		13963	33$\bar{1}$	10113	10108	31$\bar{2}$	9929	9925	33$\bar{1}$		9650
			051	11258	11249	33$\bar{1}$		9928	032		9661
			41$\bar{1}$	11743	11752	122	10253	10260	122	9896	9896
			321	11898	11905	241	12082	12078	330	9982	9983
			132	12068	12073	212	12726	12719	051	10616	10622
			241		12082	042		12725	311		10637
			042	12774	12759	340	12884	12887	32$\bar{2}$	10711	10720
			34$\bar{1}$		12794	420	13653	13658	15$\bar{1}$		10725
			25$\bar{1}$	13061	13058				41$\bar{1}$	11362	11358
			24$\bar{2}$	13323	13329						
			420	13722	13726						
			20$\bar{3}$	14228	14222						
			41$\bar{2}$		14238						
			003	14881	14891						

Table 2 lists the observed $\sin^2\theta$ values of the measured reflexions, together with the calculated values.

The crystals were prepared by Mrs. H. Zandbergen of the Kamerlingh Onnes Laboratorium and the X-ray photographs were taken by Mr. A. Verhoorn.

References

FISHER, D. J. (1957). *Z. Kristallogr.* **109**, 73.
GROTH, P. (1908). *Chemische Krystallographie.* Vol. II, p. 525. Leipzig: Engelmann.
KLUG, H. P. & ALEXANDER, L. E. (1940). *J. Amer. Chem. Soc.* **62**, 1492.
WOLFF, P. M. DE (1948). *Acta Cryst.* **1**, 207.

Acta Cryst. (1964). **17**, 780

Paramètres et groupes spatiaux du chlorodurène et du bromodurène.

Par G. CHARBONNEAU, J. BAUDOUR, J. CL. MESSAGER et J. MEINNEL, *Groupe de Recherches de physicochimie structurale, Faculté des Sciences de Rennes, France*

(*Reçu le* 22 *janvier* 1964)

Les mesures diélectriques ont montré que de nombreux dérivés pentasubstitués du benzène présentaient du polymorphisme. Nous avons entrepris l'étude des structures de différentes phases du chloro-1-tétraméthyl-2,3,5,6-benzène (monochlorodurène) et du bromo-1-tétraméthyl-2,3,5,6-benzène (monobromodurène). Nous avons déterminé leurs paramètres et groupes spatiaux par des diagrammes d'oscillation, de rotation et de Weissenberg. Les mesures de densité ont été faites par flottation dans des solutions de nitrate d'argent.

Monochlorodurène

Par évaporation lente de solution dans l'éthanol à 20 °C, nous avons obtenu des plaques allongées suivant la direction [010]. Le système est monoclinique:

$$a=15{,}89\pm0{,}05,\quad b=5{,}80\pm0{,}02,\quad c=11{,}17\pm0{,}04\ \text{Å};$$
$$\beta=109°7'\pm30'$$

Densité mesurée: 1,15 Densité calculée: 1,153
Groupe spatial $P2_1/a$ $Z=4$.

Monobromodurène

Forme β — Par évaporation d'une solution d'éthanol à 45 °C, nous avons obtenu des cristaux en forme de plaques allongées suivant la direction [010], ces cristaux scellés dans un tube capillaire restent sous cette forme à l'état métastable à 20 °C; le système est monoclinique:

$$a=16{,}07\pm0{,}05,\quad b=5{,}80\pm0{,}02,\quad c=11{,}39\pm0{,}04\ \text{Å},$$
$$\beta=110°45'\pm30'$$

Densité mesurée: 1,42 Densité calculée: 1,426
Groupe spatial $P2_1/a$ $Z=4$.

Forme α — L'évaporation lente des solutions dans l'éthanol à 20 °C fournit des cristaux sous forme d'aiguilles dont l'axe est dans la direction [010]. La système est orthorhombique et à 20 °C:

$$\mathrm{a}=14{,}62\pm0{,}05,\quad b=5{,}43\pm0{,}02,\quad c=12{,}05\pm0{,}04\ \text{Å}$$

Densité mesurée: 1,47 Densité calculée: 1,478
Groupe spatial $P2_12_12_1$ $Z=4$;

à 40 °C il se transforme en forme β.

Acta Cryst. (1964). **17**, 781

Dispersion corrections and crystal structure refinements*.

By James A. Ibers and Walter C. Hamilton, *Chemistry Department, Brookhaven National Laboratory, Upton, New York, U. S. A.*

(*Received* 9 *December* 1963 *and in revised form* 16 *January* 1964)

Patterson (1963) has recently considered the treatment of the effects of anomalous dispersion in X-ray crystal structure refinements. He indicates that there are many ways of taking these effects into account and that he holds no particular brief with the way he has chosen, namely to correct the *observed* structure amplitudes for both least-squares and Fourier refinements. We feel that such a correction when used in the least-squares refinement has disadvantages, and we advocate instead the inclusion of the dispersion effects in the *calculated* structure factors.

The notation is that of Patterson. Let there be one element in the structure that shows dispersive effects; the generalization to several elements is obvious. Let this element have a geometrical structure factor H_d+iK_d and atomic scattering factor $f_d+\Delta f_d'+i\Delta f_d''$. Let the total contributions of the nondispersive elements to the structure factor be A_n+iB_n. Further define

$$\begin{aligned}A&=A_n+f_dH_d=A_n+A_d\\ B&=B_n+f_dK_d=B_n+B_d\,.\end{aligned}$$

Thus A and B are the nondispersive structure factor components for the whole structure. Let + indicate the structure factor for the plane (hkl) and − that for ($\bar{h}\bar{k}\bar{l}$). Then we have

$$\begin{aligned}F_c=F_\pm=(A+\Delta f_d'H_d-\sigma\Delta f_d''K_d)&\\ +i(\sigma B+\sigma\Delta f_d'K_d+\Delta f_d''H_d)&\quad(1)\end{aligned}$$

where σ is +1 for F_+ and −1 for F_-. Patterson defines an 'experimental' value of $|F|_o$, which we designate G_o, in the following way:

$$\begin{aligned}G_o^2=\tfrac{1}{2}(|F_+|_o^2+|F_-|_o^2)-2\delta_1(AA_d+BB_d)&\\ -(\delta_1^2+\delta_2^2)(A_d^2+B_d^2)&\quad(2)\end{aligned}$$

where $\delta_1=\Delta f_d'/f_d$ and $\delta_2=\Delta f_d''/f_d$ and where $|F_+|_o^2$ and $|F_-|_o^2$ are the observed values of $|F_+|^2$ and $|F_-|^2$. Patterson suggests that the simplest procedure is to use this 'experimental' G_o instead of $|F|_o$ both in the least-squares refinement and as a coefficient in the Fourier series to obtain an 'observed' electron density map. In the latter application the appropriate phase angle α is taken to be that which is calculated in the absence of dispersive effects, namely $\tan\alpha=A/B$. The disadvantages of this approach are as follows: (1) As Patterson points out, it is necessary in the final stages of refinement to recalculate G_o after each cycle of least-squares. In effect the assumptions concerning the model are distributed between G_o and F_c. (2) In order to apply the method of equation (2) to a non-centrosymmetric crystal both $|F_+|_o^2$ and $|F_-|_o^2$ must be observed; if they are both observed, then it is appropriate to include them separately in the observational equations (perhaps with different weights) rather than to average them first.

A straightforward approach to the problem which avoids these difficulties is to base the analysis directly on the usual $|F|_o$ and on $|F_c|$ of equation (1)†. In this way all of the observations can be included with proper

---

* Research performed under the auspices of the U.S. Atomic Energy Commission.

† Such a procedure, which has been employed here for some time, can be carried out by making minor modifications to existing least-squares programs, such as the Busing–Levy ORFLS program.