macroscopic means. Several measurements of the thermal expansion of metals using X-ray diffraction were made during the decade 1930–9, and for the most part the values agreed, within limits of experimental error, with the macroscopic values. The few cases of disagreement may have been due to the use of impure metals or to faulty temperature determination.

In a careful investigation on three alkali halides, however, Gott (1942) reported that the lattice expansion of pure potassium iodide, as measured by X-rays, was about 15% less than the macroscopic expansion in the range 20–190° C. A difference of this magnitude, if it exists, should result in such strains in the crystal as to produce obvious physical changes and would necessitate an extremely high degree of imperfection of the single crystal at elevated temperatures. As a possible explanation Gott suggested the difference between the surface layers of the crystal, which are effective in X-ray diffraction, and the internal structure of the crystal which results from Schottky false structure. In the Schottky picture, alkali and halogen ions leave their lattice positions and migrate to the surface of the crystal in numbers which increase with increased temperature. At the surface they take up new lattice positions, with the lattice points at the surface almost completely filled. However, the extent of this defect structure, which can be studied by a number of methods including electrical conductivity, alteration of density, and color-center phenomena, apparently is insufficient to produce differences of the magnitude observed by Gott.

Measurements have been made in this laboratory in an attempt to provide further information on this point. Two of the alkali halides reported on by Gott (KI and KBr) have been studied here. We are not able to support the discrepancy found by Gott; our values for the lattice-expansion coefficient show excellent agreement with the macroscopic values. Our measurements on both substances were made using two different types of high-temperature cameras, using two indirect and one direct method of temperature determination. One camera, a cylindrical powder camera with an electrically-heated furnace, has been described previously (Connell, 1947). In its use temperatures were measured both by calibration of the furnace, using known polymorphic transition points, and by the same standard-substance-admixture method used by Gott. The other camera consisted of a back-reflection cassette and pinhole system from a General Electric XRD unit and a simple liquid-bath furnace with a blade heater which was shown by test to provide direct temperature control of the powder sample.

Our measurements on KI were made using Fe $K\alpha$ radiation, instead of Cu $K\alpha$ as used by Gott, because of the superior back-reflection lines obtained with iron radiation. Two sets of back lines are produced, the (640) α_1 line at about 81° Bragg angle, and the (711) (551) α_1 line at 78° at room temperature. Measurements of the α_1 and α_2 lines from both sets of planes gave four sensitive back-region lines for measurement. KI samples were prepared from C. P. Baker's Analyzed KI of purity greater than 99·95%.

Lattice expansion of KBr was measured using both iron and copper radiations. With Fe $K\alpha_1$ radiation the (622) line falls at about 77° at room temperature, and with Cu $K\alpha_1$ the (660) (822) line falls at about 82°. The KBr was C. P. Baker's Analyzed, purity 99·99%.

The average expansion coefficients determined by us, together with the values calculated from Gott's results, are shown in Table 1. Our values represent an average of the results from diffraction patterns taken in both cameras. These results differed by less than 1%, but because of possible systematic error in the reading of the rather broad back-reflection lines, we estimate 2% as the probable accuracy of these values. The values shown for Gott's results are calculated from a curve plotted from his published data.

Table 1. *Average thermal expansion coefficients for* KI *and* KBr

Substance	Temp. range (° C.)	Our X-ray value ($\times 10^{-6}$)	Gott X-ray value ($\times 10^{-6}$)	Gott macroscopic value ($\times 10^{-6}$)
KI	20–100	40·6	37·2	40·8
	20–190	42·1	36·1	42·7
KBr	18–100	40·5	38·8	40·8
	18–190	41·7	40·5	41·4

It is seen that our values are in rather close agreement with Gott's macroscopic values and the difference between them is less than the combined limits of error.

Because of uncertainty in the interpretation of their results, we have not included in Table 1 the values reported by Eucken & Dannohl (1934) for the macroscopic expansion of these two substances. They do not define their 'true' expansion coefficient, but apparently mean $dl/l_{20}dt$ rather than $dl/l_t dt$. In either case we are unable to agree with Gott's interpretation of their results as agreeing closely with his own macroscopic values. Since it thus appears that there may be disagreement as to macroscopic expansivity also, we plan to make further measurements of both macroscopic and lattice expansion on the same crystalline sample.

References

Connell, L. F. (1947). *Rev. Sci. Instrum.* **18**, 367.

Eucken, A. & Dannohl, W. (1934). *Z. Elektrochem.* **40**, 814.

Gott, A. (1942). *Ann. Phys., Lpz.*, **41**, 520.

Acta Cryst. (1951). **4**, 76

A preliminary X-ray investigation of dodecahydrotriphenylene, $C_{18}H_{24}$, and perhydrocoronene, $C_{24}H_{36}$.

By F. Halla and W. R. Ruston, *Association pour les Études Texturales, 4 rue Montoyer, Brussels, Belgium*

(*Received* 21 *October* 1950)

In the course of an extended research program on hydrogenated aromatic hydrocarbons with high molecular symmetry, the following structural data have so far been obtained:

(1) *Dodecahydrotriphenylene*, $C_{18}H_{24}$

Space group C_{6v}^4–$C6mc$ or D_{6h}^4–$C6/mmc$, $I_a = 5{\cdot}91$, $I_c = 7{\cdot}63$ A., volume of the unit cell $V = 230{\cdot}5$ A.^{3}. Using the density averaged from literature data (Egloff, 1947), $d_4^{20} = 1{\cdot}145$ g.cm.$^{-3}$, the number of molecules in the unit cell is $n = 0{\cdot}663$ or $\doteqdot \frac{2}{3}$. This fractional number is the result of a statistical occupation of the individual homologous points in a closest packing of spheres or rotational ellipsoids in the sense of a one-dimensional disorder (Jagodzinski, 1949 *a*, *b*, *c*). This disorder becomes apparent from the continuous streaks along layer lines of the first and the second kind, streaks which are characteristic for these structures. A full account of this phenomenon will be published later in collaboration with H. Jagodzinski.

(2) *Perhydrocoronene*, $C_{24}H_{36}$

The hexagonal unit cell has the dimensions $I_a = 10{\cdot}27$, $I_c = 5{\cdot}00$ A. and $V = 458$ A.^{3}. Using the approximate density, $d \doteqdot 1{\cdot}08$ g.cm.$^{-3}$, the number of molecules in the unit cell is $n = 1$.

References

EGLOFF, G. (1947). *Physical Constants of Hydrocarbons.* New York: Reinhold.

JAGODZINSKI, H. (1949*a*). *Acta Cryst.* **2**, 201.

JAGODZINSKI, H. (1949*b*). *Acta Cryst.* **2**, 208.

JAGODZINSKI, H. (1949*c*). *Acta Cryst.* **2**, 298.

Acta Cryst. (1951). **4**, 77

X-ray crystallography of cyanamide, H_2NCN.

By C. L. CHRIST,* *Research Laboratory, American Cyanamid Co., Stamford, Connecticut, U.S.A.*

(*Received* 14 *August* 1950)

Crystals of cyanamide, suitable for X-ray study, are rather difficult to obtain, owing to the fact that cyanamide transforms readily in the presence of traces of moisture to the dimer dicyandiamide. The material used in this study was kindly supplied by Dr D. Kaiser, of this Laboratory. It consisted of a large crystalline aggregate, which had developed in an *o*-dimethylphthalate solution of the material, over a period of several years. A small fragment was detached from the mass of material and sealed into a small-diameter, thin-walled pyrex capillary together with some of the mother liquor. During this manipulation care was exercised to ensure that the crystal fragment did not come into direct contact with the atmosphere.

A preliminary rotation pattern showed that a principal crystallographic direction lay almost parallel with the axis of rotation of the capillary, thus furnishing a convenient crystal rotation axis. Weissenberg patterns and a rotation pattern were taken about this axis (subsequently designated the *b* axis) with nickel-filtered copper radiation ($\lambda = 1{\cdot}542$ A.). Measurements on the rotation pattern yielded the following unit cell dimensions:

$$a = 9{\cdot}03 \pm 0{\cdot}02, \quad b = 7{\cdot}06 \pm 0{\cdot}02, \quad c = 6{\cdot}82 \pm 0{\cdot}01 \text{ A.}$$

Reflections of the type $0kl$ occur only with k even, $h0l$ only with l even, and $hk0$ only with h even. These data are consistent with the space group *Pbca*, in which the general positions are eight-fold. Assuming eight H_2NCN per unit cell, the calculated density is 1·282 g.cm.$^{-3}$. The measured density (20° C.) is 1·282 g.cm.$^{-3}$.

---

* Present address: U.S. Geological Survey, Washington 25, D.C., U.S.A.

Acta Cryst. (1951). **4**, 77

Examination of two alkalimonofluochromates.

By RICHARD BØGVAD and A. H. NIELSEN, *The Chemical and Mineralogical Laboratories of 'Kryolitselskabet Øresund A/S', Copenhagen, Denmark*

(*Received* 19 *October* 1950)

The previously known compounds

(I) $KCrO_3F$ and (II) NH_4CrO_3F

(Werner, 1895; Hoffmann, 1912–14, vol. 2, pp. 529, 536; Olsson, 1927) were produced by treating $K_2Cr_2O_7$ and $(NH_4)_2Cr_2O_7$ with hot dilute hydrofluoric acid in excess, followed by evaporation. After standing at room temperature, the crystals were recrystallized from diluted hydrofluoric acid. In this way beautiful, well-developed crystals were obtained: the ammonium compound as ruby-red needles as large as 23 mm. in length, the potassium compound as red to orange-coloured crystals. The chemical analyses were performed on fresh, air-dried material with the following results:

(I)	Found	Cr, 32·8 %	K, 24·7 %	F, 11·4 %
	Calc.	32·89	24·73	12·02

0·0899 g. was treated with hydrochloric acid and KI. The titration required 16·9 ml. of a $Na_2S_2O_3$ solution (0·1009 N).

(II)	Found	Cr, 37·4 %
	Calc.	37·96

0·0830 g. required 17·7 ml. of a $Na_2S_2O_3$ solution (0·1009 N).

$KCrO_3F$. Tetragonal; specific gravity: 2·66 (Ketelaar & Wegerif (1938): 2·72; Beck (1940): 2·66); refractive indices: $\epsilon = 1{\cdot}644$, $\omega = 1{\cdot}622$; optically positive.

NH_4CrO_3F. Orthorhombic; specific gravity: 2·22; refractive indices: $\alpha = 1{\cdot}623 \pm 0{\cdot}002$, $\gamma = 1{\cdot}639 \pm 0{\cdot}002$; $2V = 64° \pm 2°$; optically positive.

A. H. Nielsen has carried out the synthetic and analytical work. Richard Bøgvad has made the determinations of the optical data and of the specific gravities.

References

BECK, G. (1940). *J. Prakt. Chem.* **156**, 237.

HOFFMANN, M. K. (1912–14). *Lexikon der anorganischen Verbindungen.* Leipzig: Barth.

KETELAAR, J. A. A. & WEGERIF, E. (1938). *Rec. Trav. chim. Pays-Bas*, **57**, 1269.

OLSSON, F. (1927). Dissertation, Uppsala.

WERNER, A. (1895). *Z. anorg. Chem.* **9**, 407.