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Sodium chlorate: precise dimensions for the ClO_3^- ion.

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Least-squares refinement of the structure of $NaClO_3$ with an extensive set of Mo $K\alpha$ photographic intensities has yielded precise dimensions for the ClO_3^- ion and a description of its motion in this crystal. The ion librates nearly isotropically with a r.m.s. amplitude of 5°. After correction for libration, the bond length is 1·502 (3) Å and the bond angle 106·8 (2)°.

The structure of $NaClO_3$ ($P2_13$, $Z = 4$) was first determined by Zachariasen (1929). It was later refined two-dimensionally by Aravindakshan (1959); Zachariasen (1965) published a refinement based on a limited set of 99 reflections measured with Cu $K\alpha$ radiation, in connection with a study of extinction effects and radiation damage. The present study, completed (except for the thermal-motion analysis) before Zachariasen's (1965) publication, was undertaken to provide precise dimensions for the ClO_3^- ion.

A crystal, 0·3 mm on edge, was obtained by recrystallization of AR grade $NaClO_3$ from water. Non-integrated photographic intensities were collected for layers $h = 0$ to 6 by the multiple-film equi-inclination Weissenberg technique with Zr-filtered Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å). The intensities were estimated visually and scaled internally by comparison of equivalent reflections on different layers. The differences in intensities that were observed for equivalent reflections were random and smaller than 10%, indicating that no significant crystal decomposition had occurred during the 250 h of exposure. Of 473 accessible unique reflections, 409 had intensities above background. Because 020, 120, and 111 were badly affected by extinction, they were omitted from the refinement. No absorption corrections were made [μ(Mo $K\alpha$) = 12·7 cm^{-1}]; the maximum difference in F^2 because of absorption was less than 10%.

The structure was refined anisotropically by full-matrix least squares, with Hughes's (1941) weighting scheme and $a = 6{\cdot}575$ Å at 23 °C (Deshpande & Mudholker, 1960). The final R was 0·045 for 406 observed reflections;† the calculated values for all 'unobserved' reflections were less than $1{\cdot}5F_{\min}$. Neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1962); no correction was made for anomalous dispersion. The final positional and thermal parameters are in Table 1. The thermal motion of the ClO_3^- ion was analyzed (Schomaker & Trueblood, 1968); the ion librates nearly isotropically, with r.m.s. amplitudes of 5° about the threefold axis and two axes normal to it (Table 2). Translational motion is comparatively small. An analysis of Zachariasen's (1965) data gave similar results for **L** (although the e.s.d.'s were three times larger),

Table 1. *Final atomic parameters with estimated standard deviations in parentheses*

The dimensions of U^{ij} are Å^2.

Na	x	0·0683 (7)	O	x	0·3034 (4)
	U^{11}	0·0236 (11)		y	0·5931 (4)
	U^{12}	−0·0020 (13)		z	0·5053 (4)
				U^{11}	0·0247 (9)
Cl	x	0·4182 (2)		U^{22}	0·0214 (9)
	U^{11}	0·0146 (4)		U^{33}	0·0317 (11)
	U^{12}	−0·0011 (4)		U^{12}	0·0035 (8)
				U^{13}	0·0036 (7)
				U^{23}	−0·0055 (8)

Table 2. *Eigenvalues of* **L**, **T**, *and* **S** *for the chlorate ion*

$^r\mathbf{T}$ is the reduced **T** of Schomaker & Trueblood (1968) [equations (11) and (20)], and **S** is the symmetrized **S** [equation (12)]. For each tensor, the eigenvector of the unique eigenvalue is along [111] and the other two are normal to this direction

L	26 (1) deg^2	$^r\mathbf{T}$	0·0157 (3) Å^2	**S**	0·081 (17) deg Å
	25 (1)		0·0157 (3)		−0·041 (17)
	25 (1)		0·0124 (3)		−0·041 (17)

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† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32586 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

but the elements of **T** were about twice as great, because of the displacive radiation damage in his crystal.

The Cl–O length before correction for libration is 1·490 (3) Å; correction for libration increases this to 1·502 (3) Å. The bond angle, essentially independent of the librational motion, is 106·8 (2)°. The intra-ionic O–O distance is 2·411 Å after correction for libration, and the distance of the Cl atom from the plane of the three O atoms is 0·559 Å. Zachariasen's (1965) data lead to libration-corrected geometry very similar to that found in the present study. These parameters for the ClO_3^- ion accord well with those found for other Cl–O species (Wells, 1975).

The shortest Na···O distances are 2·50 and 2·54 Å and the shortest Na···Cl distance is 3·98 Å.

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N-(*N*-Piperidylacetyl)piperidinium perchlorate: correction of a printer's error.

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In Jaskólski, Gdaniec & Kosturkiewicz [*Acta Cryst.* (1977), B**33**, 1627–1630] three lines of text on p. 1628 have been transposed. The second paragraph of the *Discussion* should begin: 'The geometry of the perchlorate anion is given in Table 4. The Cl–O distances are considerably shorter than the accepted value of 1·46 Å (Truter, Cruickshank & Jeffrey, 1960). The numerous peaks . . .'

All the relevant information is given in the Abstract.

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Structural studies of incipient pentacoordination of silicon in hydrido transition-metal silyl compounds. I. The crystal structure of *cis*-hydridotriphenylsilyl(η-cyclopentadienyl)dicarbonylrhenium (η^5-C_5H_5)-$Re(CO)_2HSi(C_6H_5)_3$: correction of printer's errors.

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Errors introduced in Smith & Bennett [*Acta Cryst.* (1977), B**33**, 1113–1117] prior to final printing are corrected. The β angle given in the Abstract should read 92·18 (8)°; the density quoted is the observed value.

All the relevant information is given in the Abstract.