

Table 2. *Interionic distances (Å) and bond angles (°) in Li<sub>2</sub>MnCl<sub>4</sub>*

Li(2)—Cl	4 ×	2.391 (1)	Cl—Li(1), Mn—Cl	180.00 (0)
Li(1), Mn—Cl	6 ×	2.560 (1)	Cl—Li(1), Mn—Cl	86.94 (4)
Cl—Cl	3 ×	3.523 (3)	Cl—Li(1), Mn—Cl	93.06 (4)
Cl—Cl	6 ×	3.7159 (1)	Cl—Li(2)—Cl	109.47 (6)
Cl—Cl	3 ×	3.904 (3)	Li(1), Mn—Cl—Li(1), Mn	92.98 (4)
Li(1), Mn—Li(1), Mn	6 ×	3.7134 (0)	Li(1), Mn—Cl—Li(2)	123.13 (5)
Li(1), Mn—Li(2)	6 ×	4.3544 (0)		

dehydration in a stream of dry HCl gas in steps up to 500°C and 350°C respectively.

Stoichiometric amounts of the starting materials were fused together in dry nitrogen atmosphere after an evacuation up to 10<sup>-5</sup> torr; thereupon they were annealed during one week just below the melting point.

The X-ray powder diffraction patterns were obtained by means of a Philips PW 1050 diffractometer, modified for the investigation of hygroscopic samples. The compounds in question appeared to have cubic symmetry; systematic absences indicated a face-centred structure. Intensity calculations, carried out for both the inverse and the normal spinel structure, suggested that the compounds Li<sub>2</sub>TCl<sub>4</sub> (T = Mg, Mn, Fe, Cd) have the inverse spinel structure. This means that half of the lithium ions are tetrahedrally surrounded by chlorine ions whereas the other half, together with the T ions, are distributed statistically over the occupied octahedral sites (Wyckoff, 1964).

A structure refinement of Li<sub>2</sub>MnCl<sub>4</sub>, based on neutron diffraction powder data, confirmed our conclusions. The neutron diffraction diagram was collected at 300 K on the powder diffractometer at the Petten High Flux Reactor. The complete range of data (5.4° < 2θ < 138.7°) obtained with a wavelength of 2.5718 Å was used in the refinement. Absorption and extinction effects were small and no corrections were made. The sample, about 20 g, was contained in a thin-walled vanadium tube. The scattering lengths (10<sup>-12</sup>) were -0.194 for Li, -0.360 for Mn and 0.960 for Cl (Neutron Diffraction Commission, 1969). The function minimized during the least-squares analysis of the powder diffraction profile (Rietveld, 1967) was  $\sum w[y(\text{obs}) - (1/c)y(\text{calc})]^2$ ;  $y(\text{obs})$  and  $y(\text{calc})$  represent the observed and calculated intensities per measuring point in a peak,  $w$  is the statistical weight and  $c$  the scale factor. A few cycles of full matrix refinement led to convergence at  $R_1 = 100 \sum |I(\text{obs}) - (1/c)I(\text{calc})| / \sum I(\text{obs}) = 2.59$ ,  $R_2 = 100 \sum [y(\text{obs}) - (1/c)y(\text{calc})] / \sum y(\text{obs}) = 6.36$ ,  $R_3 = 100 / \{ \sum w [y(\text{obs}) - (1/c)y(\text{calc})]^2 / \sum w [y(\text{obs})]^2 \} = 6.70$ .

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### The crystal and molecular structure of bis[diethyl bis-(1-pyrazolyl)borato]nickel(II): a correction. By H. M.

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In Fig. 2 of Echols & Dennis [*Acta Cryst.* (1974). B30, 2173–2176], the bond distance N(1)–C(3) should read 1.34 instead of 1.39.

The correction of a typographical error: In the paper on bis[diethyl bis-(1-pyrazolyl)borato]nickel(II) by Echols & Dennis (1974), Fig. 2, the bond distance N(1)–C(3) should read 1.34 instead of 1.39. All other bond lengths and angles are printed correctly.

The final scale factor and overall temperature factor were 0.0411(4) and -1.92(6). Inclusion of a site occupation number resulted in reliability indices  $R_1 = 1.90$ ,  $R_2 = 6.38$  and  $R_3 = 6.53$ .

All structural data of interest are gathered in Table 1. Standard deviations, given in parentheses, are based on counting statistics. Interionic distances and bond angles are presented in Table 2.\*

The computations were carried out partly at the Computing Division of the Reactor Centre in Petten and partly at the Computing Institute of Leiden University. We wish to thank the staff of the R. C. N. in Petten. One of us (J. de J.) is especially grateful for an allowance provided during a six-month stay. The investigations were supported by the Netherlands Foundation for Chemical Research (S.O.N.) with financial aid from the Netherlands Organization for the Advancement of Pure Research (Z. W. O.).

\* A list of intensities has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31174 (2 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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