Thermochimica Acta, 121 (1987) 203-214
Elsevier Science Publishers B.V., Amsterdam – Printed in The Netherlands

EFFECT OF THE DIFFERENT STRUCTURAL DISTORTIONS OF MoO_3 AND THE HOMOLOGOUS $Mo_{18}O_{52}$ UPON THE THERMODYNAMICS OF Li^+ INSERTION *

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(Received 30 October 1986)

ABSTRACT

The thermodynamics of Li^+ insertion in two layered oxides, MoO_3 and $Mo_{18}O_{52}$, has been evaluated by electrochemical and calorimetric techniques. The two compounds have similar structures, but the distortion of the first, especially in the plane of the layers, is significantly larger. This causes a structural rearrangement for MoO_3 which gives rise to high integral entropies of insertion per mole of Li^+. $\Delta S/x$ values of 80–110 J K^{-1} mol^{-1} have been calculated in the range $0 < Li^+/mol < 1.0$.

$Mo_{18}O_{52}$ shows $\Delta S/x$ values within the limits expected for a reaction between solid components, thus indicating a lower degree of structural disturbance induced by Li^+.

INTRODUCTION

The thermodynamics of Li^+ insertion in the crystalline structures of several oxides of interest in Li cells have recently received attention. From the enthalpic and entropic terms found at various insertion levels for each compound, the Li^+ binding energies and information on the degree of structural disturbance undergone by the oxides can be obtained [1–3]. In a recent investigation on some Mo and V oxides, some had high integral entropies of solution per mole of Li^+ ($\Delta S/x$) [2,3]. In particular, $(Mo_{0.3}V_{0.7})_2O_5$ and MoO_3 showed high values ($\Delta S/x > 100$ J K^{-1} mol^{-1} at $Li^+/mol = 0.5$) which were considered to be due to the structural reorganization brought about by Li^+ uptake.

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* Dedicated to Professor W.W. Wendlandt on the occasion of his 60th birthday.

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The calorimetric technique has proven rather sensitive to revealing differences, with respect to Li^+ insertion, between apparently similar structures. This was the case for V_2O_5 and $(Mo_{0.3}V_{0.7})_2O_5$ [2,3], whose $\Delta S/x$ values differed by ~ 180 J K^{-1} mol^{-1} in spite of the similar arrangement of the octahedra upon which their structures are based [4].

With this premise, a thermodynamic investigation of Li^+ insertion in the layered oxides MoO_3 and $Mo_{18}O_{52}$ (the latter having the basic structure of the former [5]) seemed sensible. Previously, we had limited ourselves to investigating a single composition for MoO_3 ($Li_{0.5}MoO_3$). In the present study, the two Mo oxides are compared in a wide composition range (0.1–1.2 Li^+/mol).

EXPERIMENTAL

MoO_3 was a high-purity product used without further purification, apart from drying. $Mo_{18}O_{52}$ (which will be referred to as $MoO_{2.89}$ in this paper) was prepared according to Kihlborg's technique [6] and its X-ray powder pattern matched that reported by this author.

Chemical lithiation of these materials was accomplished with a 0.4 M BuLi solution in hexane. The oxides were stirred for 14 days with proper amounts of this solution. The lithiated materials were then equilibrated for 10 days in $LiClO_4$–PC solutions. The composition of the lithiated samples was directly checked by a chemical titration [7].

The calorimetric technique has already been described [3]. For the electrochemical tests, a 1 M $LiClO_4$–PC solution was used in prismatic cells with the oxides pressed on a nickel screen. The X-ray analyses were made with the use of Ni filtered CuK_α radiation.

RESULTS

The thermodynamic cycle used for calculating Li^+ insertion in MoO_3 is based on the following reactions.

(a) Partial molar enthalpy, $\Delta\overline{H}_1$, of the reaction of solid Li_xMoO_3 in $K_3Fe(CN)_6^{3-}$ and KOH solution:

$$Li_xMoO_{3(s)} + x Fe(CN)^{3-}_{6(sol)} + 2OH^-_{(sol)}$$
$$= xLi^+_{(sol)} + MoO^{2-}_{4(sol)} + xFe(CN)^{4-}_{6(sol)} + H_2O_{(sol)} \tag{1}$$

(b) Partial molar enthalpy, $\Delta\overline{H}_2$, of pure water in the same solution:

$$H_2O_{(l)} = H_2O_{(sol)} \tag{2}$$

(c) Partial molar enthalpy, $\Delta\overline{H}_3$, of LiOH, yH_2O solution in $K_3Fe(CN)_6$ and KOH solution:

$$(\mathrm{LiOH},\ y\mathrm{H_2O})_{(l)} = \mathrm{Li}^{+}_{(sol)} + \mathrm{OH}^{-}_{(sol)} + y\mathrm{H_2O}_{(sol)} \tag{3}$$

(d) Partial molar enthalpy, $\Delta\overline{H}_4$, for the reaction of solid MoO_2 in $K_3Fe(CN)_6$ and KOH solution:

$$\mathrm{MoO}_{2(s)} + 2\mathrm{Fe(CN)}^{3-}_{6(sol)} + 4\mathrm{OH}^{-}_{(sol)} = \mathrm{MoO}^{2-}_{4(sol)} + 2\mathrm{Fe(CN)}^{4-}_{6(sol)} + 2\mathrm{H_2O}_{(sol)} \tag{4}$$

(e) Partial molar enthalpy, $\Delta\overline{H}_5$, for the solution of solid MoO_3 in KOH and $K_3Fe(CN)_6$ solution:

$$\mathrm{MoO}_{3(s)} + 2\mathrm{OH}^{-}_{(sol)} = \mathrm{MoO}^{2-}_{4(sol)} + \mathrm{H_2O}_{(sol)} \tag{5}$$

The symbols (l) and (s) indicate liquid and solid pure substances, respectively, while (sol) denotes compounds in solution.

A linear combination of the $\Delta\overline{H}_1$–$\Delta\overline{H}_5$ values

$$\Delta\overline{H}_6 = \Delta\overline{H}_1 + x(y+0.5)\Delta\overline{H}_2 - x\Delta\overline{H}_3 - x/2\Delta\overline{H}_4 - (1-x/2)\Delta\overline{H}_5 \tag{6}$$

gives the following equation

$$\begin{aligned}&\mathrm{Li}_x\mathrm{MoO}_{3(s)} + x(y+0.5)\mathrm{H_2O}_{(l)}\\ &\quad = x/2\mathrm{MoO}_{2(s)} + (1-x/2)\mathrm{MoO}_{3(s)} + x(\mathrm{LiOH},\ y\mathrm{H_2O})_{(l)}\end{aligned} \tag{7}$$

By combination of the measured values of eqn. (7) with the standard enthalpies of formation (Table 1) of the remaining components, standard enthalpies of formation are obtained for Li_xMoO_3.

Using these values, the enthalpy for lithium insertion can be written as the difference between the molar standard enthalpies of formation

$$x\mathrm{Li(s)} + \mathrm{MoO_3(s)} = \mathrm{Li}_x\mathrm{MoO_3(s)} \tag{8}$$

For the $Li_xMoO_{2.89}$ bronze the following reaction was used:

$$\begin{aligned}&\mathrm{LiMoO}_{2.89} + (0.22+x)\mathrm{Fe(CN)}^{3-}_{6(sol)} + 2.22\mathrm{OH}^{-}_{(sol)}\\ &\quad = \mathrm{MoO}^{2-}_{4(sol)} + (0.22+x)\mathrm{Fe(CN)}^{4-}_{6(sol)} + x\mathrm{Li}^{+}_{(sol)}\end{aligned} \tag{9}$$

TABLE 1

Molar enthalpies of formation of various compounds at 298.16 K (kJ mol^{-1})

Compound	ΔH_f^0	Ref.
$H_2O_{(l)}$	-285.85 ± 0.04	8
LiOH, $52.91H_2O$	-506.7 ± 1.3	8
$MoO_{2(s)}$	-588.94 ± 1.05	18
$MoO_{3(s)}$	-745.05 ± 0.77	18
$MoO_{2.89(s)}$	-715.55 ± 0.80	this work

TABLE 2

Partial molar enthalpies of reaction and solution from eqns. (2)–(5) (kJ mol^{-1})

	This work	Literature values [8]
ΔH_2^0	-0.07 ± 0.01	-0.079 ± 0.002
ΔH_3^0		-2.13 ± 0.03
ΔH_4^0	-273.60 ± 0.50	-293.62 ± 0.58
ΔH_5^0	-85.95 ± 0.40	-83.83 ± 0.10

which, combined with eqns. (2)–(5) gives

$$\Delta \overline{H}_{10} = \Delta \overline{H}_1 + x(y + 0.5)\Delta \overline{H}_2 - x\Delta \overline{H}_3 - (0.11 + x/2)\Delta \overline{H}_4 \\ - \left[1 - (0.11 + x/2)\Delta \overline{H}_5\right] \qquad (10)$$

hence one obtains

$$\mathrm{Li}_x\mathrm{MoO}_{2.89} + x(y + 0.5)\mathrm{H_2O}_{(\mathrm{l})} \\ = (0.11 + x/2)\mathrm{MoO}_{2(\mathrm{s})} + \left[1 - (0.11 + x/2)\mathrm{MoO}_{3(\mathrm{s})}\right] + x(\mathrm{LiOH},\ y\mathrm{H_2O})_{(\mathrm{l})} \qquad (11)$$

from which the standard enthalpies of formation of $\mathrm{Li}_x\mathrm{MoO}_{2.89}$ are obtained. These values allow the insertion enthalpies of the following reaction to be calculated:

$$x\mathrm{Li}_{(\mathrm{s})} + \mathrm{MoO}_{2.89} = \mathrm{Li}_x\mathrm{MoO}_{2.89} \qquad (12)$$

The enthalpy of formation for $\mathrm{MoO}_{2.89}$ was obtained by eqns. (10) and (11), where x was set equal to zero.

In the various reactions, concentrations less than 10^{-3} m were used, therefore the $\Delta \overline{H}$ values were assumed to be equal to ΔH^0 [2,3]. All the values obtained from eqns. (1)–(12) are reported in Tables 2, 3 and 4. While the ΔH_2^0 and ΔH_3^0 values determined in this work are very close to literature values [8], ΔH_4^0 differs significantly with respect to Dickens et al.'s

TABLE 3

Molar enthalpy of reaction, formation and insertion of $\mathrm{Li}_x\mathrm{MoO}_3$ at 298.16 K (kJ mol^{-1})

x	ΔH_1^0	ΔH_7^0	ΔH_8^0
0.1	-93.05 ± 0.50	-775.79	-30.69
0.2	-101.10 ± 0.40	-805.54	-60.43
0.3	-109.14 ± 1.20	-835.28	-90.18
0.5	-130.42 ± 0.90	-889.59	-144.49
0.8	-168.74 ± 1.30	-964.64	-219.54
1.0	-195.61 ± 0.80	-1013.35	-268.25
1.2	-220.36 ± 1.00	-1064.18	-319.08
1.7	-318.20 ± 1.10	-1155.30	-410.19

TABLE 4

Molar enthalpies of reaction, formation and insertion of $Li_xMoO_{2.89}$

x	ΔH_9^0	ΔH_{11}^0	ΔH_{12}^0
0.1	-129.90 ± 0.50	-741.78	-26.23
0.2	-141.89 ± 0.80	-767.58	-52.03
0.3	-154.91 ± 0.90	-792.35	-76.80
0.5	-182.10 ± 0.70	-840.74	-125.19
0.8	-226.12 ± 1.20	-910.10	-194.54
1.0	-254.93 ± 0.50	-956.86	-241.31
1.2	-295.11 ± 0.60	-992.27	-276.72

data [8]. We have no explanation for this difference. However, the impact of this discrepancy on the intercalation enthalpies is greatly attenuated by the fact that ΔH_4^0 only appears in one of the five terms of eqn. (6). Therefore, the difference in the ΔH_8^0 values is not remarkable. For example, for the Li^+ intercalation in MoO_3 at $x = 0.2$, one obtains 60 and 62 kJ mol^{-1} by the present work and using literature values, respectively.

Equations (8) and (12) give the standard enthalpies of insertion in the Li_xMoO_3 and $Li_xMoO_{2.89}$ phases at various x values, while $\Delta H_8^0/x$ and $\Delta H_{12}^0/x$ represent the integral enthalpies of solution per mole of Li^+ in the oxides. At least five experiments related to processes (1), (2), (4) and (5) were made, and the standard error of the mean was calculated. The uncertainty for a thermochemical cycle is the root of the sum of the variances of each term. The uncertainties on the integral insertion enthalpies per mole are ~ 2 kJ.

As reference states, pure MoO_3 and $MoO_{2.89}$ for the solvent and $x \rightarrow 0$ for the solute were assumed.

The integral free energies of insertion per mole of Li^+ ($\Delta G/x$, where ΔG is obtained by integration of the E/x curves) and the related integral

TABLE 5

Integral free-energy and enthalpy values per mole of Li^+ inserted in MoO_3 at 298.16 K (kJ mol^{-1})

x	$\Delta G/x$	$\Delta H_8^0/x$
0.1	-275.43	-306.93
0.2	-274.61	-302.17
0.3	-271.89	-300.61
0.5	-262.21	-288.97
0.8	-250.46	-274.42
1.0	-245.74	-268.25
1.2	-241.93	-265.90
1.7	-236.56	-241.29

TABLE 6

Integral free energy and enthalpy values per mole of Li^+ inserted in $MoO_{2.89}$ at 298.16 K (kJ mol^{-1})

x	$\Delta G/x$	$\Delta H_{12}^0/x$
0.1	−254.68	−262.28
0.2	−250.58	−260.14
0.3	−247.13	−256.00
0.5	−242.72	−250.38
0.8	−234.79	−243.18
1.0	−232.79	−241.31
1.2	−224.10	−230.60

TABLE 7

Comparison between literature values for $\Delta H^0/x$ of Li_xMoO_3 and the values from this work at the same x (kJ mol^{-1})

x	Literature values	This work
0.40	−287 [10]	−295
0.54	−284 [1], −290 [9]	−287
0.93	−253 [1,9]	−270
1.74	−239 [1], −243 [9]	−236

enthalpies (per mole of Li^+), $\Delta H^0/x$, are reported in Tables 5 and 6. For MoO_3 some $\Delta H^0/x$ values have also been measured by Dickens and Reynolds [1,9,10]. In Table 7 a comparison between the data of these authors and the present data for the same x values is made. While it is not known to us why these authors report slightly different values at $x = 0.54$ and $x = 1.74$ in two different papers [1,9], fair agreement is found between their data and those of this work, apart from the enthalpy at $x = 0.93$.

The $\Delta G/x$, $\Delta H_8^0/x$ and $\Delta H_{12}^0/x$ values of Tables 5 and 6 were used to calculate $-\Delta S/x$ with the equation

$$-\Delta S/x = (\Delta G/x - \Delta H^0/x)/T \qquad (13)$$

DISCUSSION

MoO_3 is usually reported as having a layered structure built up of octahedra at two levels [11]. In Fig. 1a, for example, the octahedra drawn with heavy lines are at different levels and their edge-sharing produces zig-zag rows along the [001] direction. The octahedral coordination of MoO_3 is, however, so highly distorted that it affords an alternative description in terms of a chain structure based on tetrahedra (Fig. 1b).

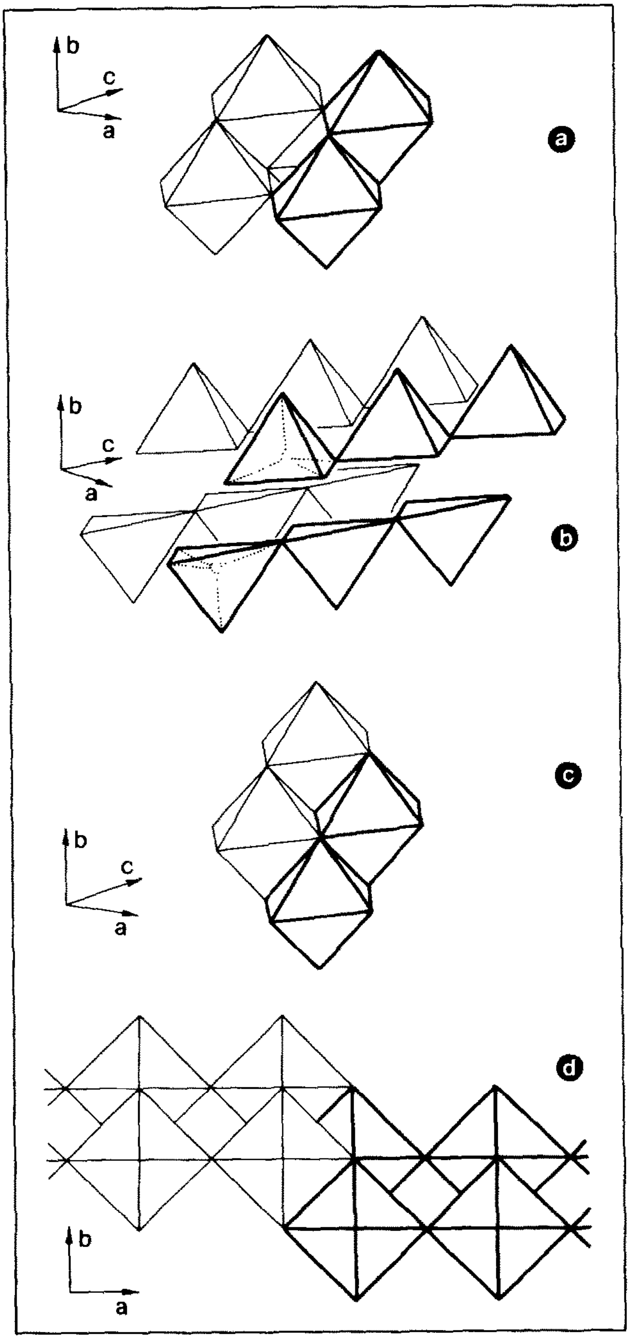

Fig. 1. (a) Idealized structure of MoO_3 based on octahedra; (b) alternative structure of MoO_3 based on tetrahedra; (c) octahedra shearing in $Mo_{18}O_{52}$; (d) zig-zag rows of octahedra in $Mo_{18}O_{52}$.

$Mo_{18}O_{52}$ has a structure of the MoO_3-type. Using the common octahedral model, both oxides can be regarded as formed by layers cut into strips of finite width. One may think that one layer in $Mo_{18}O_{52}$ derives from one layer in MoO_3 by a shear mechanism, regarding octahedra lying on the borderlines of the layer strips [11]. An example of this shearing is shown in Fig. 1c where the octahedra with heavy lines have sheared so as to share edges with the octahedra with faint lines. The resulting layers are regularly stepped and the zig-zag rows of octahedra have the shape shown in Fig. 1d. The oxygen deficiency implied by the formula $MoO_{2.89}$, with respect to MoO_3, is restricted to the regions where a shearing has occurred.

The distances between Mo atoms of octahedra sharing edges are 0.3–0.6 Å shorter than those of Mo atoms of octahedra sharing corners. Therefore, a structure with a greater number of edge-sharing octahedra is less favourable from an electrostatic viewpoint. On this basis the ideal structure of $Mo_{18}O_{52}$ should be less stable than that of MoO_3. However, the real structure of the former is such that one Mo atom at each end of every zig-zag row has a tetrahedral coordination. Therefore, the number of short Mo–Mo distances exceeds the equivalent number in MoO_3 by only one.

On the other hand, by comparing the unit cell dimensions of MoO_3 with those of the basic structure of $Mo_{18}O_{52}$ one obtains:

MoO_3: $a = 3.96$ Å, $b = 13.85$ Å, $c = 3.70$ Å

$Mo_{18}O_{52}$: $a = 3.84$ Å, $b = 14.46$ Å, $c = 3.73$ Å

For both, b is twice the interlayer distance, while a and c represent Mo–Mo distances in the layer planes. It can be seen that in $Mo_{18}O_{52}$ a and c have closer values, i.e. there is a considerably reduced distortion with respect to MoO_3 in the direction corresponding to [100]. Furthermore, the Mo–O distances are quite similar in the plane of the zig-zag rows, but the values for the two bonds perpendicular to this plane are more nearly equal in $Mo_{18}O_{52}$ than in MoO_3. Mo–O is equal to 1.73 and 2.25 Å for MoO_3 and 1.80 and 2.12 Å for $Mo_{18}O_{52}$, respectively [11].

Therefore, even though both structures have to be considered as distorted, this characteristic is certainly less important for the latter. Distortion plays a major role with respect to the features of Li^+ insertion. Besenhard et al. [12] have observed for MoO_3 a maximum around 0.5 Li^+/mol for the variation of the interlayer distance ($b/2$) during discharge. The lower values for higher contents of intercalated Li^+ require, according to these authors [12], a structural rearrangement within the MoO_3 layers. Indeed, the unit cell dimensions reported above change, as measured by Dickens and Reynolds [1], to $a = 3.64$ Å, $b = 16.7$ Å and $c = 3.77$ Å for an $Li_{0.5}MoO_3$ sample (chemical lithiation). The Mo–Mo distances within the layers have become more similar in this lithiated material than in the Li-free oxide. A stabilizing effect on the distorted structure of MoO_3 may therefore be assigned to Li^+.

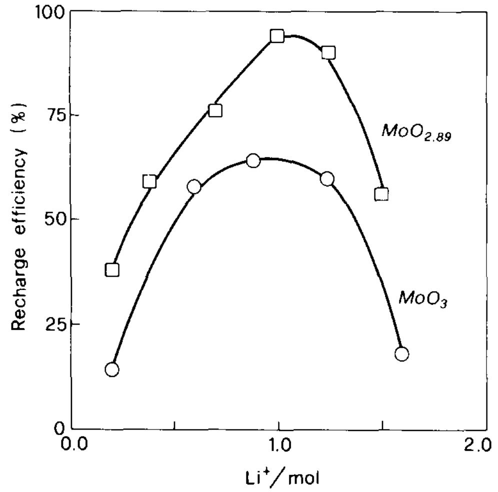

Fig. 2. Recharge efficiency (first cycle) as a function of depth of discharge for MoO_3 and $MoO_{2.89}$ electrodes at 1 mA cm^{-2} in $LiClO_4$-PC.

This is confirmed by the evidence that, upon recharge, it is impossible to recover all Li^+ inserted [13,14]. For shallow discharges, i.e. 0.1–0.3 Li^+/mol, the recharge efficiency is particularly poor due to Li^+ remaining in the structure. As for the non-stoichiometric oxide its ability to release Li^+ is clearly superior, with a maximum efficiency of 95% (Fig. 2). The existence of a certain level of distortion in this oxide is indicated by the incomplete recharge at low insertion values.

The coulometric titration curves (Fig. 3) also have quite different profiles for the two materials. For MoO_3, a peculiar shape is observed [1,15], with a short initial pseudo-plateau followed by a steep decline preceding a second long pseudo-plateau. $Mo_{18}O_{52}$ shows a more regular, although stepped, titration curve, this also indicating a less severe structural reorganization. Incidentally, we have to consider the possibility of attaining values greater

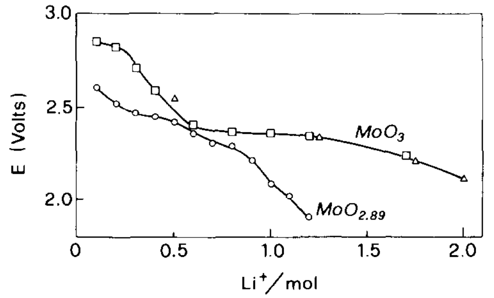

Fig. 3. Coulometric titration curves for MoO_3 and $MoO_{2.89}$ in $LiClO_4$-PC (△, Hunger and Ellison's data [15]).

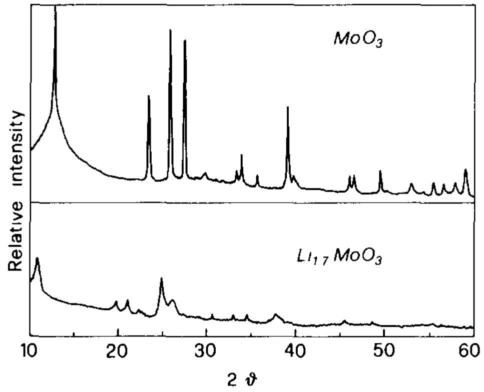

Fig. 4. X-ray patterns of MoO_3 and $Li_{1.7}MoO_3$. CuK_α radiation.

than 1.5 Li/mol for MoO_3 intercalation, which was previously reported as the maximum [13,16]. Both Hunger's [15] and our data show that 2 Li^+ can be inserted per molecule. However, too high Li^+ contents, as expected, cause great modifications in the lattice. This is clearly demonstrated in Fig. 4 by the X-ray pattern of an $Li_{1.7}MoO_3$ sample obtained by chemical lithiation. One cannot expect from the latter a good reversibility of the process of Li^+ insertion. Indeed, as shown in Fig. 2, a dramatic fall of the recharge efficiency occurs for $Li^+/mol > 1.3$, this matching previous data [13]. For $Mo_{2.89}$, a qualitatively similar bell-shaped curve is obtained, but the efficiencies are generally higher, also in the region corresponding to a high Li^+ content.

The thermodynamic data obtained by calorimetric measurements enhance the differences between the two oxides as far as the Li^+ insertion process is concerned. In Tables 5 and 6 the integral free energies per mole and the corresponding enthalpies of insertion are reported. As pointed out by Dickens and Reynolds [1], the $\Delta H^0/x$ values represent a measure of the binding energies of Li in the host oxides. From the evidence so far collected from electrochemical data, it is not surprising that such energies are larger for MoO_3. The unusually high $\Delta H^0/x$ recorded for Li^+-inserted MoO_3 provoke departures from the corresponding $\Delta G/x$ values and, hence, fairly high $\Delta S/x$ values are obtained. These are shown in Fig. 5, together with the corresponding values for $MoO_{2.89}$. In the $0 < Li^+/mol < 1.0$ range, $\Delta S/x$ shows a linearly decreasing trend for MoO_3. This can possibly be referred to a tendency of the structure to compensate for the structural rearrangement produced by the initial amounts of Li^+. Such an hypothesis is in keeping with the observed maximum for the interlayer spacing upon Li^+ insertion [12]. The layers are not rigid and tend to modify so as to accommodate the foreign ion with reduced structural disturbance. Li^+ insertions larger than 1.0 Li^+/mol cause a more rapid decrease in $\Delta S/x$. At $Li^+/mol = 1.7$, a

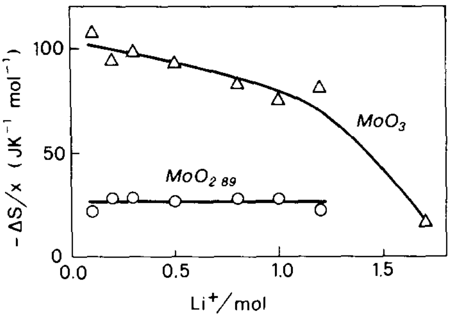

Fig. 5. Integral entropy values per mole of Li^+ inserted in MoO_3 and $MoO_{2.89}$.

value of only 20 J K^{-1} mol^{-1} was calculated. However, it is known that on deep discharge the range for the formation of a single phase is exceeded [12] and at least two phases are formed [12,16]. The last piece of evidence is confirmed by the X-ray pattern of Fig. 4. In these conditions, one cannot strictly speak of Li^+ intercalation, the overall reaction being more complicated. Therefore, the thermodynamic data of these concentrations are not so meaningful as those obtained at lower x.

For $MoO_{2.89}$, the low and constant (~ 25 J K^{-1} mol^{-1}) $\Delta S/x$ value testifies the relative ease of Li^+ accommodation in its structure.

CONCLUSION

The thermodynamics of Li^+ insertion in the structurally similar oxides MoO_3 and $MoO_{2.89}$ have shown remarkable differences in both the electrochemical and the calorimetric approaches. The high level of distortion of the MoO_3 structure generates an entropic term which does not merely correspond to the configurational entropy of insertion [17]. A significant additional contribution comes from its structure rearrangement. As the shape of the electrochemical titration curve also depends on the entropy of insertion [17], the peculiar profile for MoO_3, and its difference from that of $Mo_{18}O_{52}$, can be accounted for.

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