# Sulphide Capacities of CaO-Al<sub>2</sub>O<sub>3</sub> Slags in the Temperature Range 1773–1848 K.

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## **ABSTRACT**

The sulphide capacities of CaO-Al<sub>2</sub>O<sub>3</sub> binary slags of nine different compositions were experimentally determined at 1773 and 1848 K using the gas-slag equilibration method. From the results obtained, the sulphide capacities and the molar sulphide capacities have been calculated. The results are compared with data reported in the literature. Comparisons of the experimental Cs values with values calculated on the basis of optical basicity concept are presented. The experimental molar sulphide capacities are correlated with Darkens excess stabilities for the system CaO-Al<sub>2</sub>O<sub>3</sub>. A theoretical model for prediction of the sulphide capacity developed in the Department of Theoretical Metallurgy has been used to describe the variation of C<sub>s</sub> as a function of temperature and composition, in the case of CaO-Al<sub>2</sub>O<sub>3</sub> slags.

## 1. INTRODUCTION

Fincham and Richardson /1/ proposed that the sulphur-oxygen exchange reaction between gas and slag phase be described as

$$1/2 S_2(gas) + O^{2}(slag) = S^{2}(slag) + 1/2 O_2(gas)$$
 (1)

These authors defined the sulphide capacity of a slag, C<sub>s</sub>, in terms of the partial pressures of sulphur and oxygen in the gas phase as

$$C_S = (Wt - \% S \text{ in slag}) \cdot (p_{O_2} / p_{S_2})^{1/2} = K_1 \cdot (\frac{a_{O^2}}{\gamma_{S^2}})$$
 (2)

where  $K_1$  is the equilibrium constant for reaction (1),  $a_{\mathrm{O}^{2-}}$  is the activity of oxygen ions and  $\gamma_{\mathrm{S}^{2-}}$  , the activity coefficient of S2- ions in the slag. The Cs values for different slags can be determined experimentally by equilibrating the same with a gas mixture of known p<sub>S</sub>, and p<sub>O</sub>, and analysing for sulphur content. Using this classical method, the values of C<sub>S</sub> for a wide range of slags have been experimentally determined at the temperatures of interest in metallurgy. Such measurements have been reviewed earlier by Kärsrud /2/ as well as Young et al. /3/. A comparison of the experimental results of different sulphide capacity determinations is rendered difficult in view of the fact that the experiments have been carried out under widely differing conditions of temperature and  $(p_{O_2}/p_{S_2})$ ratio. In the case of complex slags, the compositions could be very different. In order to make a meaningful comparison of different experimental results, it is necessary to have a theoretical model that could enable reliable extrapolations of the data as functions of temperature and composition. In this respect empirical equations like optical basicity could often lead to in uncertainties estimations. This has demonstrated in the case of the sulphide capacities of CaO-MnO-SiO<sub>2</sub> slags in a previous work /4/ carried out in this laboratory. A theoretical model has earlier been developed in the Division of Theoretical Metallurgy at the Royal Institute of Technology /5/ that would enable the estimation of the sulphide capacities of complex

slags from those of lower order systems. The application of the model presupposes the availability of reliable experimental data for the sulphide capacities of simpler systems. The present experimental determination was carried out aiming to provide reliable data for the above model.

#### PREVIOUS WORK

The sulphide capacities of CaO-Al<sub>2</sub>O<sub>3</sub> binary slags have been determined by a number of workers in the past. A direct comparison of the results, however, is difficult due to the variation in the experimental temperature as explained earlier. In the present work, the model calculations were made on the basis of the present as well as earlier experimental data. It should be pointed out that the earlier published data usually involve uncertainties in the  $p_{s_2}/p_{o_2}$  ratios calculated for various gas mixtures. The Gibbs energies of formation of the various species in the CO-Co<sub>2</sub>-SO<sub>2</sub>-Ar mixtures at high temperatures have been accurately determined in recent years and are found to be different from earlier data. Consequently, the  $p_s$ ,  $/p_o$ , ratios calculated from the relative amounts of the various gases in the gas mixtures have been found /3/ to be very different from those estimated earlier, sometimes as high as 50%, especially when  $(p_{s_2}/p_{o_2})^{1/2}$  is higher than 1000. In the absence of raw data, the recalculation of earlier results would be impossible and, hence, the

choice is restricted to experiments with  $(p_{s_2}/p_{o_2})^{1/2}$  < 500.

In Table 1, the sources and the slag compositions from literature used for comparison with the results obtained in the present work are shown. It is to be noted that, in the case of the measurements of the sulphide capacities by Carter and Macfarlane /6/, two slag compositions containing 58 and 57 wt% Al<sub>2</sub>O<sub>3</sub> (denoted as CA1 and CA2 by the authors) may probably be in the two phase region as can be seen in the phase diagram available today /12/, reproduced in Fig. 1.

### **EXPERIMENTAL PROCEDURES**

### Materials

The experimental method used is based on the equilibrium between a gas mixture consisting of CO-CO<sub>2</sub>-SO<sub>2</sub>-Ar and a liquid slag at a given temperature according to equation (1). The materials used in the present work are listed in Table 2.

The powders of Al<sub>2</sub>O<sub>3</sub> and CaO were dried, heated to 1273 K in air, held at that temperature for 10 hours, rapidly cooled and stored in a desiccator. After cooling, they were finely ground in an agate mortar, weighed and mixed in the required proportion. Five grams of each powder mixture was prefused at 1873 K in a platinum crucible. The prefused slags were ground to fine sizes in an agate mortar and stored in a desiccator.

Table 1
Previous work used for the present assessment

Reference	Authors	T (exp) [K]	X <sub>ALO3</sub>	$(p_{S_2} / p_{O_2})^{1/2}$
[6]	Carter & Macfarlane	1773	0.285-0.402	<358
[7]	Richardson&Fincham	1923	0.333-0.500	<500
[8]	Kor & Richardson	1773	0.337-0.402	<500
[9]	Hino,Kitag.&Ban-Ya	1823, 1923	0.274-0.454	<106
[10]	Zhao & Reddy	1773, 1923	0.302-0.5	<500
[11]	Cameron at al.	1823	0.277	<450

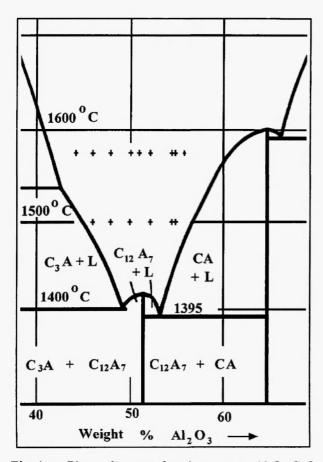


Fig. 1: Phase diagram for the system Al<sub>2</sub>O<sub>3</sub>-CaO /12/, with the experimental slag compositions marked.

CA: Al<sub>2</sub>O<sub>3</sub>·CaO, C<sub>3</sub>A: Al<sub>2</sub>O<sub>3</sub>·3CaO, C<sub>12</sub>A<sub>7</sub>: 7Al<sub>2</sub>O<sub>3</sub>·12CaO.

## **Apparatus**

The furnace assembly as shown in Fig. 2 is described elsewhere /2,13/. The gas mixtures used for equilibration with the slag were purified by the gascleaning trains shown in Fig. 3. The flow rates of individual gases were controlled and monitored using BRONKHORST HI-TEC mass flow meters/controllers, model number F 201C-FA, connected to a four-channel digital readout and control system FLOW-BUS. The flow rate of the gas mixture was maintained at approximately 400 ml/min during all the experiments. The equilibrium partial pressures of oxygen and sulphur in the gas phase at the experimental temperature and pressure were calculated by the Gibbs energy minimization program SOLGASMIX /14/. The sulphur in the slag was analyzed by the stoichiometric combustion method /15/. METROHM automatic titration apparatus was used to carry out the redox titration in accordance with the procedure described earlier /13/.

## RESULTS

Equilibration experiments for CaO-Al<sub>2</sub>O<sub>3</sub> slags were carried out at 1773 and 1848 K. The values of  $(p_{s_2}/p_{o_2})^{1/2}$  were 360 and 151.3. Each experiment was repeated at least once at the given temperature and gas composition and the results were reproducible. The

Table 2				
Materials used in the present study				

Material	Purity	Supplied By:
Aluminum Oxide, (Al <sub>2</sub> O <sub>3</sub> )	Anhydrous, AR grade	Fisher Scientific, New Jersey, U.S.A.
Calcium Oxide, (CaO)	Anhydrous, AR grade	Fisher Chemical, New Jersey, U.S.A.
Argon, (Ar) GAS	Argon plus	AGA Gas, Stockholm
Carbon Monoxide, (CO)	S grade	AGA Gas, Stockholm
Carbon Dioxide, (CO <sub>2</sub> )	S grade	AGA Gas, Stockholm
Sulphur Dioxide, (SO <sub>2</sub> )	S grade	AGA Gas, Stockholm

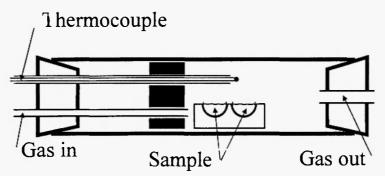


Fig. 2: The experimental setup.

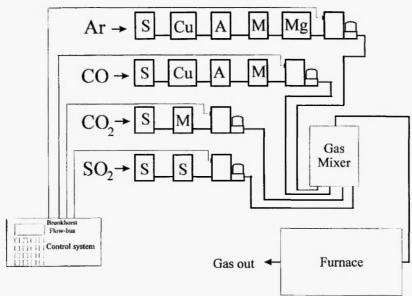


Fig. 3: Gas purification train.

S' silica gel. A' ascarite. M' magnesium perchlorate. Cu' copper turn

S: silica gel, A: ascarite, M: magnesium perchlorate, Cu: copper turnings at 773 K and Mg: magnesium flakes at 773 K.

slag compositions are indicated in the  $CaO-Al_2O_3$  phase diagram /12/ in Fig. 1 and are well within the homogeneous liquid region. The compositions of the slags used are also presented in Table 3 in weight % as well as mole fraction, together with the basicity ratio. The  $p_{s_2}$  and  $p_{o_2}$  values, wt% S obtained by chemical analysis and the sulphide capacity values are presented in Table 4. All slags have been completely melted during equilibration which was confirmed by microscopic observations after every experiment.

In order to avoid considering the differing atomic weights of the components and the effect of the same, Abraham et al. /16/ expressed the sulphide capacities in

terms of the mole fractions of sulphide, thus defining the term molar sulphide capacity, C'<sub>s</sub> as

$$C_{S'} = \left(\frac{n_{S}}{n_{CaO} + n_{Al_2O_3}}\right) (p_{O_2} / p_{S_2})^{1/2}$$
 (3)

where n, is the number of moles of component i.

This concept gives a direct insight into the correlations between the structure of the slag and its sulphur absorbing power. The values of C', calculated from the results obtained in the present work are also presented in Table 4.

Sample name	Wt - % Al <sub>2</sub> O <sub>3</sub>	Wt - % CaO	Х <sub>м,о,</sub>	X <sub>cao</sub>	$\frac{X_{cao}}{X_{Al_2O_3}}$	Wt-% CaO Wt-% Al <sub>1</sub> O <sub>3</sub>
Slag A	56.0	44.0	0.4117	0.5883	1.429	0.785
Slag B	55.0	45.0	0.402	0.598	1.487	0.818
Slag C	54.5	45.5	0.397	0.603	1.5189	0.835
Slag D	52.2	47.8	0.375	0.625	1.6666	0.916
Slag E	51.0	49.0	0.364	0.636	1.747	0.960
Slag F	50.0	50.0	0.354	0.646	1.824	1.00
Slag G	47.9	52.1	0.3358	0.6642	1.978	1.087
Slag H	46.0	54.0	0.319	0.681	2.134	1.174
Slag I	44.1	55.9	0.3026	0.6974	2.304	1.267

Table 3
Compositions and basicities of the slags used in the present work

## DISCUSSION

Sulphide capacities of slags have been found to vary with basicity and several empirical relationships have been proposed. Sosinsky and Sommerville /17/ have applied the concept of optical basicity, which is a measure of the electron donor power of oxides, to slags, taking into account even the effect of temperature. They derived an expression valid in the temperature range 1673-1973 K, viz,

$$Log C_s = (22690-54640 * \Lambda)/T + 43.6* \Lambda - 25.2$$
 (4)

where  $\Lambda$  is the optical basicity of the slag.

Recently, Young et al. /3/ made a re-evaluation of the C<sub>S</sub> dependency on the optical basicity, using multiple regression techniques. They demonstrated that separate nonlinear correlations are required for different ranges of optical basicity. The equation they arrived at includes the concentration of some oxide components in the slag as well as the temperature and is presented below:

$$Log C_S = -13.913 + 42.84*\Lambda - 23.82*\Lambda^2 - (11710/T)$$
  
-0.02223 \* (Wt-% SiO<sub>2</sub>) - 0.02275 \* (Wt-%Al<sub>2</sub>O<sub>3</sub>)  
when  $\Lambda$ < 0.8

The optical basicity in the above equation is calculated using empirical coefficients for other oxide components in the slag proposed by previous authors /17/. A comparison of the calculated C<sub>S</sub> values based on eqns. (4) and (5) with the values obtained in the present work is shown in Table 5. It is seen that the agreement between the sulphide capacities calculated using eqns. (4) and (5) and the experimental data can at best be considered as reasonable. It should be kept in mind that the concept of optical basicity is highly empirical and its correlation with the sulphide capacity is rather complex for most slags used in iron and steelmaking.

The experimentally obtained molar sulphide capacities,  $C'_S$  at 1773 and 1848 K, are presented as functions of composition in Fig. 4. It is seen that  $C'_S$  increases with temperature and decreases as the  $Al_2O_3$  content in the melt increases. The sulphur absorbing capacity of the slag is dependent on the availability of  $O^{2-}$  ions in the slag according to reaction (1). As the  $Al_2O_3$  content increases, the  $O^{2-}$  ions get increasingly attached to  $[Al_2O_4]^{2-}$  units according to the reaction

$$Ca^{2+} + O^{2-} + Al_2O_3 = Ca^{2+} + [Al_2O_4]^{2-}$$
 (6)

Consequently, the sulphide capacity is expected to

Table 4

The experimental parameters, sulphide capacities, molar sulphide capacities obtained in this work

Sample	Temp.	Ps,	P <sub>O</sub> ,	Wt-%S	n <sub>s</sub>	EX	PT.
	(K)	(atm)	(atm)	WI-765	705	Cs	C's
Slag A	1848	4.74e-3	2.07e-7	0.1372	3.21e-3	9.07e-4	2.12e-5
Slag B	1848	4.74e-3	2.07e-7	0.1998	4.65e-3	1.31e-3	3.07e-5
Slag B	1848	4.74e-3	2.07e-7	0.1997	4.58e-3	1.29e-3	3.02e-5
Slag B	1773	7.34e-3	5.66e-8	0.1995	4.64e-3	5.54e-4	1.288e-5
Slag C	1848	4.74e-3	2.07e-7	0.2535	5.88e-3	1.68e-3	3.884e-5
Slag C	1773	7.34e-3	5.66e-8	0.409	9.49e-3	1.14e-3	2.632e-5
Slag D	1848	4.74e-3	2.07e-7	0.1638	3.75e-3	1.08e-3	2.476e-5
Slag D	1848	4.74e-3	2.07e-7	0.1911	4.37e-3	1.26e-3	2.888e-5
Slag D	1773	7.34e-3	5.66e-8	0.188	4.30e-3	5.22e-4	1.194e-5
Slag E	1848	4.74e-3	2.07e-7	0.323	7.34e-3	2.13e-3	4.847e-5
Slag F	1848	4.74e-3	2.07e-7	0.327	7.39e-3	2.16e-3	4.879e-5
Slag F	1848	4.74e-3	2.07e-7	0.3484	7.87e-3	2.30e-3	5.200e-5
Slag F	1848	4.74e-3	2.07e-7	0.548	1.24e-2	2.82e-3	8.181e-5
Slag F	1773	7.34e-3	5.66e-8	0.524	1.18e-2	1.45e-3	3.285e-5
Slag F	1773	7.34e-3	5.66e-8	0.5585	1.26e-2	1.55e-3	3.501e-5
Slag G	1848	4.74e-3	2.07e-7	0.515	1.15e-2	3.41e-3	7.591e-5
Slag G	1773	7.34e-3	5.66e-8	0.723	1.61e-2	2.01e-3	4.477e-5
Slag H	1848	4.74e-3	2.07e-7	0.527	1.16e-2	3.48e-3	7.685e-5
Slag H	1773	7.34e-3	5.66e-8	0.927	2.05e-2	2.57e-3	5.679e-5
Slag H	1773	7.34e-3	5.66e-8	0.901	1.99e-2	2.50e-3	5.520e-5
Slag I	1848	4.74e-3	2.07e-7	0.757	1.65e-2	5.01e-3	1.092e-4

decrease. This trend is confirmed in the present results. The higher degree of dissociation at higher temperatures would lead to the increase in sulphur capacities.

It is reasonable to expect that reaction (6) would also decrease the activities of CaO and Al<sub>2</sub>O<sub>3</sub> in the melt and, consequently, decrease the integral molar Gibbs energy. In order to verify this aspect, the variation of the excess stability for the binary CaO-Al<sub>2</sub>O<sub>3</sub> system with composition was compared with the

corresponding change in the C'<sub>S</sub> values. The excess stability function, as defined by Darken /18/, can be written in this case as

$$\frac{d^2G^E}{dx_{CaO}^2} = -2RT \frac{d \ln \gamma_{CaO}}{dx_{ALO}^2} = \text{Excess Stability}$$
 (7)

where  $G^E$  is the integral molar excess Gibbs energy for the system CaO-Al<sub>2</sub>O<sub>3</sub> and  $\gamma_{CaO}$  is the activity

 $\textbf{Table 5} \\ \text{Comparison of experimental $C_S$ values with data from previous models based on the optical basicity concept}$ 

Sample	Exp. T [K]	Experimental C <sub>s</sub> ·10 <sup>-4</sup>	Ref.[17] eqn.(4) C <sub>s</sub> ·10 <sup>-4</sup>	Ref.[3] eqn.(5) C <sub>s</sub> ·10 <sup>-4</sup>
Slag A	1848	9.07	20.4	11.2
Slag B	1848	13.09	22.8	12.57
Slag B	1848	12.90	22.8	12.57
Slag B	1773	5.54	9.16	6.78
Slag C	1848	16.76	24.2	13.3
Slag C	1773	11.36	9.68	7.24
Slag D	1848	10.83	31.8	17.5
Slag D	1848	12.63	31.8	17.5
Slag D	1773	5.22	12.4	9.4
Slag E	1848	21.35	36.64	20.02
Slag F	1848	21.62	41.77	22.55
Slag F	1848	23.03	41.77	22.55
Slag F	1848	28.23	41.77	22.55
Slag F	1773	14.55	15.87	12.17
Slag F	1773	15.51	15.87	12.17
Slag G	1848	34.1	53.49	28.42
Slag G	1773	20.08	19.88	15.33
Slag H	1848	34.85	67.9	34.81
Slag H	1773	25.75	24.74	18.93
Slag H	1773	25.04	24.74	18.93
Slag I	1848	50.07	86.4	43.1

coefficient of CaO in a defined standard state. Fig. 4 illustrates the variation of excess stability with  $x_{Al_2O_3}$  at 1848 K. The corresponding activity data was taken from literature /19/. The first derivative of the molar sulphide capacity with respect to composition,  $-dC_S'/dx_{Al_2O_3}$  at the same temperature, was also plotted in the same figure. It is seen that both curves show a similarity, confirming the importance of reaction (1) in desulphurization reactions.

A modified sulphide capacity concept for binary

slags, with large differences in the basicity of the oxide components, was recently developed in the Division of Theoretical Metallurgy /20/. In this model, the sulphur in the gas is considered to react only with the most basic oxide. Since the activities of O<sup>2-</sup> and S<sup>2-</sup> cannot be directly measured, eqn. (1) may be rewritten in terms of neutral molecules dissolved in the slag, as:

$$1/2 S_2 (gas) + Me_XO (slag) = Me_XS (slag) + 1/2O_2 (gas) (8)$$

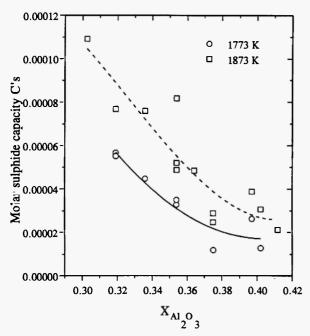


Fig. 4: Molar sulphide capacity,  $C'_S$  vs.  $x_{Al_2O_3}$ , at 1773 and 1848 K.

Expressing the equilibrium constant for the above reaction as

$$K_{8} = \left(\frac{a_{\text{Me}_{x}S}}{a_{\text{Me}_{x}O}}\right) * (p_{O_{2}} / p_{S_{2}})^{1/2} =$$

$$\left(\frac{x_{\text{Me}_{x}S} * y_{\text{Me}_{x}S}}{a_{\text{Me}_{x}O}}\right) * (p_{O_{2}} / p_{S_{2}})^{1/2}$$
(9)

and inserting the definition of C<sub>s</sub>, one gets

$$K_{8} = \left[\frac{k * Wt - \% S * \overline{W} * \gamma_{Me_{x}}}{a_{Me_{x}O}}\right] * \left(\frac{p_{O_{x}}}{p_{S_{2}}}\right)^{1/2} = \frac{k * C_{S} * \overline{W} * \gamma_{Me_{x}S}}{a_{Me_{x}O}}$$

$$(10)$$

According to this treatment, when  $\gamma_{S^{2-}}$  is independent of the ratio Me<sub>x</sub>O/Al<sub>2</sub>O<sub>3</sub>, the product of the C<sub>S</sub> and average molecular mass  $(C_S * \overline{W})$  should be proportional to the activity of Me<sub>x</sub>O. In the case of the system CaO- Al<sub>2</sub>O<sub>3</sub>, Nilsson *et al.* /20/ have plotted the C<sub>S</sub> \*  $\overline{W}$  as a function of a<sub>CaO</sub> using the literature data

and obtained a straight line at 1773 K, the slope of the line being 0.256.

A similar exercise was carried out in the present work using the present experimental results at 1773 and 1848 K along with the activity data reported by Allibert et al. /19/. This is illustrated in Fig. 6. It is seen that the straight line relationship holds for both temperatures. Another interesting aspect is that the slope at 1773 K for the present results, viz. 0.258, is very close to that obtained by Nilsson et al. /20/,

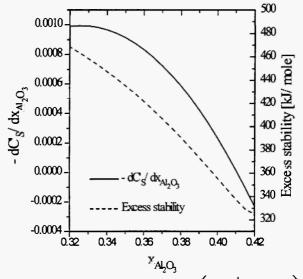


Fig. 5: Excess stability and  $\left(-dC_{S}'/dx_{Al_{2}O_{3}}\right)$ vs.  $x_{Al_{2}O_{3}}$  at 1848 K.

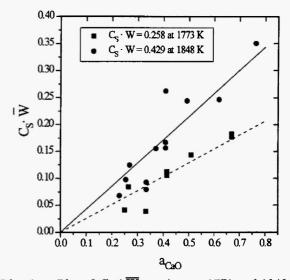


Fig. 6: Plot of  $C_S * \overline{W}$  vs.  $A_{CaO}$  at 1773 and 1848 K.

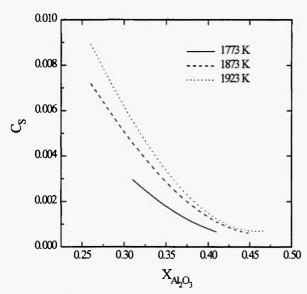


Fig. 7: Plot of  $C_8$  vs.  $x_{Al_2O_3}$ , predicted by the model at 1773, 1873 and 1923 K.

indicating that the  $C_S$  values measured in the present work are in good agreement with the earlier data.

A theoretical model for the estimation of sulphide capacities at different compositions and temperatures in the case of complex slags has recently been developed in the Division of Theoretical Metallurgy /5/. The model is based on a modified Temkin approach assuming the complete dissociation of all complex species. The model has been successfully used in the case of the CaO-MnO-SiO<sub>2</sub> system. The present results as well as the earlier results for the sulphide capacities in the case of the CaO-Al<sub>2</sub>O<sub>3</sub> system were incorporated in this model. The model predictions at 1773 and 1848 K are compared with the experimentally measured values in Table 6. It is seen that the model predictions are quite close to the experimental data confirming thereby the reliability of the model. Fig. 7 gives the model predictions at 173, 1873 and 1923 K. The increase of C<sub>s</sub> with temperature and decrease with increasing x<sub>Al<sub>2</sub>O<sub>3</sub></sub> are well brought out by the model predictions. A very interesting feature in Fig. 7 is that the Cs curve corresponding to 1923 K shows a very slight upward trend beyond  $x_{Al_2O_3} = 0.47$ . This may perhaps be attributed to the change in the role of Al<sub>2</sub>O<sub>3</sub> from an acidic to a basic oxide.

It is intended to use the present results as a basis for

Table 6
A comparison of the experimental C<sub>S</sub> with those calculated using the model for complex slags /5/

Exp. Temp. [K]	Experimental C <sub>s</sub>	Calculated C <sub>s</sub>
1848	0.00091	0.00097
1848	0.00131	0.00116
1848	0.00129	0.00116
1848	0.00168	0.00126
1848	0.00126	0.00180
1848	0.00108	0.00180
1848	0.00214	0.00212
1848	0.00282	0.00242
1848	0.00216	0.00244
1848	0.00230	0.00244
1848	0.00341	0.00309
1848	0.00349	0.00376
1848	0.00501	0.00446
1773	0.00055	0.00083
1773	0.00114	0.00091
1773	0.00052	0.00130
1773	0.00146	0.00176
1773	0.00155	0.00176
1773	0.00201	0.00223
1773	0.00250	0.00271
1773	0.00258	0.00271

model predictions of the sulphide capacities of complex industrial slags. Further experimentation regarding the sulphide capacity measurements of ternary slags containing CaO and  $Al_2O_3$  are currently in progress.

### **SUMMARY**

The sulphide capacities of CaO-Al<sub>2</sub>O<sub>3</sub> binary slags at 1773 and 1848 K were experimentally measured using the traditional gas-slag equilibration technique.

The molar sulphide capacities calculated from the experimental data were correlated with Darken's excess stability function for the CaO-Al<sub>2</sub>O<sub>3</sub> binary system. The linearity of the product  $C_S * W$  (where W is the average molecular mass) with respect to  $a_{CaO}$  was demonstrated. The results were incorporated in the theoretical model developed in the present laboratory enabling the prediction of  $C_S$  values at different temperatures and compositions.

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