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High-temperature Studies on Blastfurnace Slags.-I.

By J. E. KRÜGER, K. H. L. SEHLKE and J. H. P. VAN AARDT.*

WHEN vitreous blastfurnace slags are heated to temperatures of the order of 800 to 900 deg. C., devitrification takes place, and the heat evolved during devitrification can clearly be seen on a differential thermal analysis curve^{2,3,4,5} as an exothermal peak or as a set of exothermal peaks commencing at about 800 deg. C.; the peaks usually differ in amplitude. The devitrification of solid glassy slag and the crystallisation of molten slag were studied, in South Africa, with the aid of differential thermal analysis and X-ray diffraction methods with the object of gaining more information about the mechanism of devitrification and the hydraulic properties of various blastfurnace slags when mixed with Portland cement.

Experimental Results.

Ten samples of different slags were selected for this investigation, seven of which were South African blastfurnace slags, two were imported blastfurnace slags,

Slag No.	CaO	Al_2O_3	SiO ₂	MgO	Fe ₂ O ₃
I	28.60	21.80	28.30	18.80	0.96
2	31.00	17.00	30.40	18.00	0.20
3	36.10	12.20	35.70	13.80	0.23
4	19.00	5.00	40.00	12.00	26.70
5	41.20	16.40	35.20	4.20	0.90
6	34.20	12.80	34.60	15.10	1.66
7	32.80	12.90	34.20	17.20	o·88
8	30.20	12.70	35.20	21.00	0.42
9	36.60	13.30	34.40	13.80	0.73
10	39.20	10.60	38.10	7.80	2.87

TABLE I.—CHEMICAL ANALYSES OF SLAGS.

*The authors are in the Materials Division, National Building Research Institute, Council for Scientific and Industrial Research, Pretoria, South Afr.ca. TABLE II.—CRYSTALLINE PHASES PRESENT IN THE SLAGS HEAT-TREATED AT DIFFERENT TEM-PERATURES IN DTA-APPARATUS AND WHEN THE MOLTEN SLAG WAS COOLED SLOWLY TO ROOM TEMPERATURE. IDENTIFICATION BY X-RAY METHOD.

Slag No.	Minera to the For	al phases d temperat r key to m	etected in ures mark ineral pha	slag samp red on the ases, see f	oles heated e <i>dta</i> -curv ootnote o	l in <i>dta-</i> ap e n facing p	paratus age.	Crystal- line phases of molter slag
	a	b	c	d	e	f	g	- cooled slowly
I	Glass MA	C ₂ AS- C ₂ MS ₂ , MA, C ₃ MS ₂	C ₂ AS- C ₂ MS ₂ , MA, C ₃ MS ₂	C ₂ AS- C ₂ MS ₂ , MA, C ₃ MS ₂	$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ MA,\\ CMS,\\ \beta-C_{2}S \end{array}$	_		C ₂ AS- C ₂ MS ₂ , CMS, MA
2	Glass MA	$\begin{array}{c} C_2AS \\ C_2MS_2, \\ C_MS_2, \\ C_MS_2, \\ MA, \\ \beta \cdot C_2S \end{array}$	$\begin{array}{c} C_2AS-\\ C_2MS_2,\\ C_3MS_2,\\ MA,\\ \beta\text{-}C_2S \end{array}$	$\begin{array}{c} C_2AS-\\ C_2MS_2,\\ C_3MS_2,\\ MA,\\ \beta-C_2S\end{array}$	C ₂ AS- C ₂ MS ₂ , CMS, MA	_	_	C 2AS- C 2MS 2, CMS, MA
3	Glass	C ₂ AS- C ₂ MS ₂ , C ₃ MS ₂	$C_{2}AS-C_{2}MS_{2}, C_{3}MS_{2}, \beta-C_{2}S$	$C_{2}AS-C_{2}MS_{2},$ $C_{3}MS_{2},$ $\beta-C_{2}S$	$C_{2}AS-C_{2}MS_{2},$ $C_{3}MS_{2},$ $\beta-C_{2}S$	$\begin{array}{c} C_{2}AS-\\ C_{2}MS,_{2}\\ \beta-C_{2}S \end{array}$		$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ CMS,\\ C_{3}MS_{2},\\ \beta-C_{2}S\end{array}$
4	Glass	Fe- CMS ₂	Fe- CMS ₂	Fe- CMS ₂				Fe- CMS ₂
5	Glass C ₂ AS- C ₂ MS ₂	C ₂ AS- C ₂ MS ₂	C ₂ AS- C ₂ MS ₂ , α-CS	C ₂ AS- C ₂ MS ₂ , α-CS	C ₂ AS- C ₂ MS ₂ , α-CS	_	_	$\begin{vmatrix} C_2 AS - \\ C_2 MS_2, \\ \beta - C_2 S \end{vmatrix}$
6	Glass	C ₃ MS ₂ , C ₂ AS- C ₂ MS ₂	C ₃ MS ₂ , C ₂ AS- C ₂ MS ₂	$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ C_{3}MS_{2},\\ \beta-C_{2}S\end{array}$	$\begin{array}{c} C_2AS-\\ C_2MS_2,\\ C_3MS_2,\\ \beta-C_2S \end{array}$	$\begin{array}{c} C_2AS-\\ C_2MS_2,\\ \beta-C_2S\end{array}$		$\begin{array}{c} C_2AS-\\ C_2MS_2,\\ CMS,\\ \beta-C_2S\end{array}$
7	Glass	C ₃ MS ₂ , C ₂ AS- C ₂ MS ₂	C ₃ MS ₂ , C ₂ AS- C ₂ MS ₂	C ₂ AS- C ₂ MS ₂ , C ₃ MS ₂	$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ \beta-C_{2}S\end{array}$	$\begin{array}{c} C_2AS-\\ C_2MS_2,\\ \beta-C_2S \end{array}$		$\begin{array}{c} C_2AS-\\ C_2MS_2,\\ CMS,\\ \beta\text{-}C_2S \end{array}$
8	Glass	C ₃ MS ₂ , CMS, C ₂ AS- C ₂ MS ₂	C ₃ MS ₂ , C ₂ AS- C ₂ MS ₂ , CMS	$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ C_{3}MS_{2},\\ CMS,\\ \beta-C_{2}S \end{array}$	$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ CMS,\\ C_{3}MS_{2},\\ \beta-C_{2}S \end{array}$	_		$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ CMS,\\ \beta\text{-}C_{2}S, \end{array}$
9	Glass	C ₃ MS ₂ , C ₂ AS- C ₂ MS ₂	C ₂ AS- C ₂ MS ₂ , C ₃ MS ₂	C ₂ AS- C ₂ MS ₂ , C ₃ MS ₂	C ₂ AS- C ₂ MS ₂ , C ₃ MS ₂	C ₂ AS- C ₂ MS ₂ , C ₃ MS ₂ , CMS	C ₂ AS- C ₂ MS ₂ CMS	C ₂ AS- C ₂ MS ₂ , CMS, C ₃ MS ₂
10	Glass	$\begin{array}{c} C_{2}AS - \\ C_{2}MS_{2}, \\ C_{3}MS_{2} \end{array}$	C ₂ AS- C ₂ MS ₂ , C ₃ MS ₂	C ₂ AS- C ₂ MS ₂	C ₂ AS- C ₂ MS ₂	$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ CMS,\\ \alpha\text{-}CS \end{array}$		$\begin{array}{c} C_{2}AS-\\ C_{2}MS_{2},\\ \beta-C_{2}S\end{array}$



Fig. 1.—Dta-Thermograms for Blastfurnace Slags.



and one (slag No. 4) was not a blastfurnace slag. The chemical analyses of the slags are given in *Table I*. The South African slags had a high magnesium-oxide content in comparison with the two imported slags (Nos. 5 and 10).

To establish the appearance of the *dta*-thermograms during devitrification, the solid glassy slags were first subjected to differential thermal analysis at a heating rate of 10 deg. C. per minute to about 1,100 deg. C. The *dta*-thermograms are shown in *Figs.* 1 and 2. Once the thermogram of a slag was known, fresh samples were heated in the *dta*-apparatus at 10 deg. C. per minute, one sample being heated to a temperature immediately before the commencement of a peak, another sample to the turning point of a peak, and another to immediately after the completion of a given peak; the points are indicated by the letters a, b, c, etc., on the *dta*-thermograms in *Figs.* 1 and 2. The heating of a sample was discontinued at one of these temperatures and the sample was allowed to cool to ambient temperature. The cooled samples were X-rayed to identify the

KEY TO MINERAL PHASES: IN TABLE	II (see	facing page)	1.
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CAS	_	2 C20 ALO SIO		geblenite)
2.13		2 CaO. A1203.5102		gemenne = melilite
C,MS,	=	2CaO.MgO.2SiO ₂		akermanite f
C ₃ MS ₂		3CaO.MgO.2SiO,	-	merwinite
MA	1000	MgO.Al,O3	=	spinel
$\beta - C_{s}S$	-	$\beta = 2$ CaÕ.ŠiO,		$\dot{\beta}$ – dicalcium silicate
CMS ⁻	-	CaO.MgO.SiO ₂	=	monticellite
$\alpha - CS$	=	pseudo-DaO.SiO2	-	pseudo-wollastonite
CMS ₂	-	CaO.MgO.SiO ₂		diopside



Slag No.	Quench temperature (deg. C.)	Phase present
I	1400 1350 1340 1325 1315 1300	$\begin{array}{c} \text{Glass} + \text{spinel} \\ \text{Glass} + \text{monticellite} + \text{spinel} \\ , + , + , + , \\ , + , + , + , \\ , + , +$
2	1400 1380 1370 1360 1350 1330 1320 1300	Glass + spinel ", + "," Glass + monticellite + spinel ", + "," + "," ", + "," + "," Glass + monticellite + melilite + spinel ", + "," + "," + ","
3	1365 1350 1345 1340 1325	Glass Glass + melilite Glass + melilite + monticellite n + n + n
4	1260 1250 1240	Glass Iron-diopside
5	1400 1355 1350 1330	Glass Glass + melilite ,, + ,, ,, + ,,
6	1400 1345 1340 1320 1310 1300	Glass Glass + monticellite ,, + , ,, + , Glass + monticellite + melilite
7	1360 1350 1340 1300 1290	Glass Glass + monticellite ", + ", ", + ", Glass + monticellite + melilite
8	1410 1400 1390 1380 1360 1343 1300 1210	Glass Glass + monticellite ", + ", " ", + ", " Glass + monticellite + melilite ", + ", + ", "

TABLE III.—QUENCH DATA FOR THE SLAGS.

(Table III concluded on page 67.)

Slag No.	Quench temperature (deg. C.)	Phase present
9	1370	Glass
	1360	Glass + merwinite + melilite
	1350	,, + ,, + ,,
	1300	Glass + melilite + monticellite
10	1310	Glass
	1300	Glass + melilite
	1290	,, + ,,
	1280	,, + ,,
	1250	,, + ,,
	1200	Glass + melilite + pyroxene
	1100	, + , +, ,

TABLE III. (Concluded from page 66.)

minerals present. *Table* II gives a summary of the crystalline phases which were present in the heat-treated blastfurnace slags, as described in the foregoing, up to the temperatures corresponding to the points on the *dta*-thermograms. The phases present when the molten slags were air-cooled are also given.

The phases present at high temperatures were investigated by means of the quenching method, using a platinum-wound furnace the temperature of which could be controlled to within \mathbf{r} deg. C. The quench data are given in *Table* III. Where the crystallinity of the heat-treated quenched and air-cooled blastfurnace slag was well developed, a polarizing microscope served to supplement the X-ray analysis. A *dta*-thermogram for slag No. 6, showing the devitrification and melting peaks during heating and the solidification peaks during cooling, is shown in *Fig.* 3.

Discussion of the Results.

GLASSES.—When slags such as those under discussion are rapidly cooled or quenched, they do not crystallise but their viscosity increases so that glasses that are stable at relatively low temperatures are formed. Such a glass is generally regarded as a material having a structure intermediate between a liquid and a solid. For a glass, the order of bond or structure is of the "short-range" type, which means that the units of structure are the same as those in the crystalline form but with the difference that the arrangement of the units in a crystal is completely regular, whereas in a glass their arrangement is almost random. Glass can therefore be regarded as a potential crystalline material which lacks the energy and mobility for transformation into the ordered crystalline condition. There are two main factors which influence the crystallisation of a liquid, namely, nucleus formation and crystal growth. Bonnetti¹ pointed out that there is a definite persistency of certain melts against crystallisation, which can be explained by their high viscosity immediately preceding the formation of crystal



Fig. 3.-Dta-Thermogram for Slag No.6.

nuclei or crystal growth at the prevailing temperature. Thus, with this persistency prevailing during cooling, a glassy structure is obtained which shows many similarities to the original melt and hence has a higher internal energy than the crystalline state; it is therefore thermodynamically unstable. Bonnetti further pointed out that it would be possible, theoretically, to change over from the glassy state to the crystalline state, that is to the thermodynamically stable state, by the addition of energy which is necessary for the transformation of the system. Such a transformation, from the glassy to the crystalline state in accordance with the law of crystallisation, is known as devitrification.

DEVITRIFICATION OF THE SLAGS. —When the blastfurnace slags under consideration were heated in an air atmosphere in a differential thermal analysis apparatus, some resultant thermograms displayed simple, others complex exothermal peaks as a result of devitrification, which began at about 800 deg. C. and was completed at around 1,000 deg. C. When the devitrified slags were heated further, melting occurred and was accompanied by an endothermal peak as shown in *Fig.* 3.

THE ENDOTHERM PRECEDING THE DEVITRIFICATION PEAK.—Immediately preceding the devitrification peak, an endothermal peak appears on the *dta*-thermograms for all the slags. An X-ray examination of the samples heated to 800 deg. C. in the *dta*-apparatus showed that no apparent change of phase coinciding with this peak had occurred. (See *Table II* for the phase detected at 800 deg. C.) Slag No. 4, which is not a blastfurnace slag, showed some devitrification at 800 deg. C., but no change of phase coinciding with the endotherm at 700 deg. C. preceding its devitrification peak. The spinel in slags Nos. I and 2 at 800 deg. C. was present in the vitreous slag at ambient temperature.

It is also unlikely that this endotherm, in the case of the slags investigated, could be due to dehydration and decarbonation reactions⁵ because a thermogravimetric analysis of some of the slags did not reveal any detectable change of weight in the vicinity of the endotherm. It should be pointed out that this endotherm is never divorced from the devitrification peak, as is apparent if the thermograms are compared with the thermogram of slag No. 4 and with those cbtained by Schramli and Bonnetti⁵.

Both these authorities attributed this peak to the absorption of energy by

the system which is required to give the glass the necessary mobility to assume the ordered crystalline structure. However, it is also possible that the vitreous slag sinters at about 800 deg. C., but when some melting, accompanied by the initial endothermal reaction, sets in, the mobility of the glassy system is suddenly increased to a level where crystallisation begins spontaneously. The endotherm which accompanies the process of melting of the glass is then dominated by the heat evolved by the transformation of the unstable glass into the stable crystalline phases.

THE DEVITRIFICATION PEAK AND THE MINERALS FORMED DURING DEVITRIFICA-TION.—The next step in the investigation was to identify the minerals formed during the devitrification of the slags and to find an explanation for the variation in the devitrification peaks for the various slags.

Schramli⁵ reported that, for the slags he examined by means of X-rays, the main product of devitrification was melilite, which is a solid solution of akermanite and gehlenite (see *Table II*), and came to the conclusion that there is no relationship between the appearance of the *dta*-thermogram and the nature of the devitrification products; he expressed doubt whether a refined technique would be of assistance.

In the discussion which follows it is endeavoured to demonstrate that the method adopted by the authors, that is to subject a sample of slag, heated to a temperature corresponding to a definite point on the *dta*-thermogram, to a mineralogical analysis offers a means of explaining the appearance of the devitrification peaks which appear on *dta*-thermograms.

First, the factors influencing the form of peaks on the *dta*-thermograms should be considered. Deflections on the *dta*-thermograms of a sample are governed solely by the rate and magnitude of increase or decrease of the temperature difference between the specimen and reference material heated simultaneously in the same furnace. Therefore for any particular specimen, the number, magnitude and form of the peaks will be determined by the number of thermal changes, the rate of the changes and the magnitude of the thermal effects capable of causing a detectable difference of temperature with respect to the reference material.

It was found that, on complete devitrification, apart from small quantities of other phases, the main devitrification product was melilite, with the exception that for slag No. 4, which differs materially in chemical composition from the other slags, the main final devitrification product was iron diopside. However, as in the case of crystallisation of the molten slag during slow cooling, usually more than one phase emerges during devitrification. Some of these phases could form simultaneously, but could also form in a certain sequence during devitrification. In addition to the occurrence of different crystalline phases at different stages during devitrification, solid-phase reactions can occur with the result that some of the crystalline phases already formed disappear and new ones appear.

Merwinite appeared as the first product of devitrification in all the South African slags (Nos. 1 to 3 and 6 to 9), as well as in slag No. 10, and is regarded as the primary phase during devitrification. When the merwinite crystallises out PAGE 70

in insignificant quantities, its contribution to deflections is not detected on the *dta*-thermogram, but is masked by the dominant formation of melilite, forming immediately after the first formation of merwinite; the melilite is then responsible for a single sharp peak at about 870 deg. C.; see thermograms 1, 2, 3 and 5 in *Fig.* I. The causes of the small peaks immediately following the main devitrification peak in the case of the latter four slags have not yet been established beyond doubt, but are attributed to the transformation of merwinite to form more melilite and phases such as β -C₂S and CMS. This may also explain the absence of a peak at 950 deg. C. in the case of slag No. 4, in which no merwinite could be detected as a devitrification phase.

When merwinite crystallises out in significant quantities, as is the case for slags Nos. 6, 7, 8, 9 and 10, it is considered responsible for the first peak (deflection in the case of slags Nos. 6 and 9) at about 870 deg. C. The third peak for slags Nos. 6 and 9 at about 930 to 940 deg. C., and the second peak for slags Nos. 7 and 8 are attributed to the solid-phase reaction in which merwinite disappears to form more melilite, CMS and β -C₂S. The second peak at 890 deg. C., for slags Nos. 6 and 9, is attributed to the continuation and completion of the melilite forming simultaneously with the primary-phase merwinite.

In spite of the fact that a reasonable explanation can be offered, on the basis of the phases detected by X-ray diffraction at different stages during devitrification of the slags, for the appearance of the *dta*-thermograms, no reasonable explanation can be offered in regard to the factors determining the formation of the relative quantities of the devitrification phases or the sequence in which they form⁵. There appears, at least, to be no explanation for these observations in terms of the chemical composition of the slags alone; compare, for example, slags Nos. 3 and 9, which have almost the same chemical composition but exhibit *dta*-thermograms of entirely different appearances and give, according to X-ray identification, different devitrification products in apparently different quantities. On the other hand, the *dta*-thermograms of slags Nos. 1, 2 and 3, which have different chemical compositions, are similar in appearance and the slags also give the same major devitrification products. However, within limits there is very likely some correlation between chemical composition and the appearance of the *dta*-thermograms.

(To be concluded)

An Honour for Dr. F. M. Lea.

DR. FREDERICK M. LEA, Director of the Building Research Station, Garston, has been awarded the third Walter C. Voss Award by the American Society for Testing Materials. The award is presented annually to an engineer or scientist who has contributed notably to the knowledge in the field of building technology with emphasis upon materials.



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JULY 1964



A Rotary-kiln Lime Plant in U.S.A.

A NEW lime works owned by Messrs. Pete Lien & Sons Inc., has begun operation near Rapid City, South Dakota, U.S.A. The calcining and hydrating plant has a capacity of 175 tons per day. The firm has been involved in the lime business for many years but discontinued operation because the two small 6-tons-per-day vertical kilns then in use were obsolete and labour costs were high. The new works, which was designed by Bullock Engineering Inc., comprises a rotary-kiln with a continuous hydrator and automatic mill. A flow-chart is given in *Fig.* 1. All the buildings and many of the components are new, but the kiln and some other components have been used elsewhere. Construction began in November 1962, and the works was in full operation by March last.

Limestone.

The limestone quarry, from which also aggregates are supplied, is adjacent to the works. The stone is taken from a local geological formation known as the Minnekahta limestone, which is in a bed about 40ft. thick. It is a hard durable stone having a calcium-carbonate content in excess of 97 per cent., and is of uniform quality throughout the quarry, thus making selective quarrying unnecessary and preventing variation in the quality of the product. A typical analysis is as follows:

Insoluble residue	1.201	perc	ent.
Iron and aluminium	0.38	,,	,,
Calcium carbonate	97.18	,,	,,
Magnesium carbonate	0.71	,,	,,



Fig. 1.

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The I-in to $\frac{3}{4}$ -in. limestone is at present taken from the crushers and hauled in tandem dump-lorries a distance of about I,000ft. to a stockpile alongside a 30-ton feed-hopper. When stone is required, it is placed in the hopper by a rubbertyred loader. Eventually a 24-in. conveyor will be installed from the crusher to the stockpile at the lime works. From the feed-hopper, the stone travels on a 24-in. belt conveyor 266ft. long to the top of a 70-ft. tower. A blower removes dust and fines as the stone drops from the conveyor into a 100-ton feed-hopper, thus improving the burning process. This feed-hopper is elevated so as to make possible easier installation of a pre-heater at a later date. Stone is fed from the bottom of the hopper over a pan-type vibrating feeder which is calibrated in tons per hour. A voltage-regulator prevents any fluctuation in the rate of feed which might change the quality of the product.

Calcining.

From the vibrating feeder, the stone drops into the rotary-kiln, which was supplied by Messrs. F. L. Smidth & Co. The kiln is 162 ft. long and has an internal diameter of 8 ft. 6 in. The shell is of $\frac{3}{4}$ -in. steel plate and is lined with 6-in. refractory bricks. The kiln is supported on three tyres and is driven by a 40-h.p. variable-speed electric motor. In case of a power failure, there is also an auxiliary diesel power unit.

The kiln is fired by natural gas, the heat consumption being about 9,000,000 B.t.u. per ton of quick-lime, but it is expected that the consumption will later be reduced considerably. Burning is done at 2,300 deg. F., and the kiln exhaust temperature is under 600 deg. F. Severe weather sometimes results in the supply of natural gas being cut off; therefore the primary-air system and burner are designed to burn bunker oil fuel. Heated oil-storage tanks will be installed later.

The primary-air fan is located in the burner-house pit. Primary and secondary air passes through the kiln, through a dust chamber, the induced-draught system with manually-controlled and automatic louvres, a double multi-cone dust collector, through a 6o-h.p. high-temperature fan, and thence into the chimney.

Cooling and Subsequent Processes.

Six Smidth "Unax" integral coolers are provided. The temperature of the product is reduced to less than 200 deg. F. upon discharge from the coolers on to a stationary grizzly and then into a 7-ton hopper from which it is fed directly on to a 20-in. heat-resistant belt-conveyor which can resist temperatures up to 500 deg. F. The conveyor discharges into a bucket elevator which has 7-in. by 16-in. buckets. This and four similar bucket elevators are large enough to accommodate increased capacity in the future. The entire works is planned to facilitate expansion.

From the first bucket elevator, material can be discharged on to an outside stock-pile or into a hammermill, or it can by-pass the mill to another bucketelevator which discharges directly on to a 16-in. screw-conveyor leading to a 305-ton silo, or on to a 2-ft. 6-in. by 6-ft. inclined double-deck adjustable vibrating screen and thence to a 175-ton silo. The silos are of steel construction. Material can be taken from either silo and recirculated for crushing and screening. Each silo is provided with a 24-in. belt-conveyor connected to a reversible conveyor, so that material can be taken directly to the bulk despatching station or up a bucket-elevator to recirculate or move along another 24-in. beltconveyor to a 25-ton surge hopper from which the hydrator is fed. From the feedbelt it can also be discharged directly into a 7-ton hopper at the packing station. A vibrating feeder on the surge hopper places the material in an 8-in. screw-conveyor discharging into the hydrator which has a capacity of 10 tons per hour.

As the lime is being discharged into the hydrator, water is introduced under pressure to accelerate hydration. The Schulthess Swiss Hardinge continuous hydrator is 4 ft. 6 in. in diameter and 22 ft. long. In this plant, the water and quick-lime are mixed and discharged into another bucket elevator. Additional water is injected by spray into the hydrator stack from which milk of lime is fed back into the hydrator. This operation recaptures dust and water from the steam which is generated. From the second elevator, raw hydrate is carried by a 16-in. screw-conveyor to a 1-ton surge hopper that feeds on to an automatic Raymond mill or to one of two 250-ton hydrated-lime storage silos.

Raw hydrate is fed into the automatic mill, and is ground and classified, and then taken up into the dust collector by a 75-h.p. electric fan. The finished hydrate can be dropped into an 8-ton hopper at the packing station or taken to one of two 250-ton silos. Aerated pads are provided in the bottom of the silcs to make the material flow more easily. Air-fluidised hydrated lime can be taken from either 250-ton silo via a 16-in. screw-conveyor to a third bucket elevator, and thence via a 16-in. screw-conveyor to the bulk-hydrate hopper at the packing station.

A four-valve hydrate packer and a one-valve pebble-lime packer are located side by side in the mill building. Half of the building, which is 62 ft. wide and 142 ft. long, is open and is used for storage of lime in bags and for despatch by road or rail, which can be carried out simultaneously from four bays along one side of the building for despatch by rail, and one bay along the other side for despatch by road.

Controls and instrumentation at the burner house include the calcining-zone and draught-fan temperature recorders, an air-velocity recorder, a revolution counter, indicators showing the position of the louvres, a natural-gas consumption recorder, manual primary air and natural-gas valves with pressure gauges, kilnrotation speed control, electrical push-button controls, and indicators for all operations from the raw-stone conveyors to the finished product passing into the storage silos.

Fifty men are employed at the works; twelve operate the plant, three work in the quarry, and thirty-five are employed in connection with transportion.

The foregoing information and the diagram in Fig. 1 are abstracted from a recent number of "Pit & Quarry".

Heat-of-hydration Tests of Portland Cement.

MR. E. S. NEWMAN and MR. H. A. BERMAN, of the U.S.A. National Bureau of Standards Institute for Applied Technology, recently reviewed the computational procedures called for in eight specifications for determining the heat of hydration of Portland cement by the heat-of-solution method. Such procedures differ in their timing and selection of available data, and in the details of their methods of calculation. These matters have been analysed in detail and have been applied to replicate heat-of-solution determinations. The reproducibility and accuracy of the results were then compared. This study should be of help in developing improved specifications of tests both for rapidly dissolving and for slowly dissolving cements.

France, Italy, the United Kingdom and the U.S.A. were among the first countries to specify procedures for determining the heat of hydration of Portland





Fig. 1.

Fig. 2.

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CONCRETE PUBLICATIONS LTD. 60 BUCKINGHAM GATE, LONDON, S.W.I cement by heat-of-solution methods, that is, by finding the difference between the heats of solution of a sample of dry cement and of a sample of hydrated cement. Several other countries have now established specifications based on either the British or American procedures. In other countries an unrelated direct-hydration method is used for determining the heat of hydration. Several studies comparing pairs of computational methods have been published, but the work at the Institute was done to make a comparison of most of the common heat-of-solution In this work, eight procedures were analysed mathematically and procedures. their results compared, using as datum a heat-of-solution determination of heattreated zinc oxide, the standard substance for Portland cement calorimetry.

Three calorimeters were also compared, using Portland cement and calibrating with zinc oxide. One was an air-surrounded Dewar flask with a Beckmann thermometer (Fig. 1), and another was a Dewar flask submerged in a constant-temperature water-bath. The third was a submerged air-jacketed precision platinum calorimeter (Fig. 2) with a platinum resistance thermometer.

Pastes were prepared from each cement in duplicate, and heat-of-solution tests were made in duplicate on the dry cement and on seven-day and twenty-eightday hydrated pastes. A second series of pastes was prepared several days later and the same three series of heat-ot-solution tests made. Four determinations were thus made for each cement at each age on each of the three calorimeters, or a total of seventy-two heat-of-solution determinations for the two cements. Each determination was calculated separately by the data-selection and calculation procedures specified for each of the eight different methods. The heat-of-hydration values for each cement were calculated from data collected from the tests.

The results of these determinations were then analysed statistically to determine the effects of the methods of calculation and testing, and the type of calorimeter. The results showed that among eight methods of determining the heat of solution of Portland cement, the best reproducibility is obtained with methods in which long rating periods and reading intervals are involved, that use time constants independently of the final rating periods, and that are not restricted by a definite duration of periods of solution and rating. The greatest accuracy was obtained in those methods having solution periods longer than 20 minutes, and those methods employing rigorous heat-leakage calculations. A constant-temperature room was found to be as effective in obtaining precision as a constant-temperature water-bath for the Dewar flasks used in the specifications, but an air-jacketed platinum calorimeter in a constant-temperature water-bath is the most precise of the three types of apparatus investigated.

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"Standard Method of Test for Heat of Hydration of Portland Cement." ASTM Designation C186-55.

Revised September 1960.

The Associated Portland Cement Manufacturers Ltd.

THE following are extracts from the Chairman's statement for the sixty-fifth Annual General Meeting. Referring to business during 1963, it is stated that, thanks to good weather up to Christmas, the tonnage lost in the early part of the year, owing to the exceptionally cold winter, has been made good.

The programme of increasing the number of bulk cement plants fed by rail at major centres of consumption continues; this is being done in close collaboration with British Railways and its effectiveness may be judged from the fact the percentage of home trade despatches by rail have increased from three per cent. in 1958 to over 14 per cent. last year. This has not only lowered the cost of distribution but takes a million tons of long-distance traffic off the roads each year.

Plans for meeting the rapidly increasing demand for cement include the building of a 300,000-ton works near Stanhope in Weardale, County Durham, which is now in course of construction and is expected to be in operation early next year. Production in Northern Ireland is to be increased. Planning consent to double the output at the Westbury works has been applied for, and conversion to the semi-dry process will soon begin at the Dunstable works.

Associated companies overseas again established a new record for sales amounting to a total of 2,898,000 tons. The Australian economy throughout the year was buoyant, but unusually heavy rains affected cement production for part of the year; nevertheless the Commonwealth Portland Cement Co., Ltd., and its subsidiary, Metropolitan Portland Cement Proprietary Ltd., experienced a satisfactory year. In Victoria, the new 300,000-ton works at Waurn Ponds, near Geelong, went into production in February.

Cement remained in keen demand in New Zealand. The Golden Bay Cement Co., Ltd., and its subsidiary, Waitomo Portland Cement Ltd., remained on full output throughout the year and sales have surpassed the previous record figure. A second bulk-cement ship is under construction and will be launched shortly.

Ocean Cement & Supplies Ltd., an associate company in British Columbia, Canada, participated in the increase in trade in that province. The Peace River dam project is proceeding and the outlook is more favourable than for some years past. Canada Cement Co., Ltd., which is the largest producer in the Dominion, recorded improved results.

The subsidiary company, Malayan Cement Ltd., worked at full production throughout the year and again exceeded previous records.

Trade in Mexico improved considerably and subsidiary companies have produced satisfactory results. The modernisation of the Mixcoac works proceeds and will be completed this year, while preparations are well advanced for the installation of a new kiln to double the output of the Atotonilco works.

In the Union of South Africa, the economy is thriving and there has been a strong demand for cement throughout the year. The subsidiary company, White's South African Portland Cement Co., Ltd., which continues to show satisfactory results, is participating with two other manufacturers in a new works at Durban which recently came into production. Following upon the grant of

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JULY 1964



independence to Kenya, conditions there have become more settled. In addition to an interest in the East African Portland Cement Co., Ltd., a large share-holding has been acquired in British Standard Portland Cement Co., Ltd., in which the other partner is Cementia Holding A.G., Zurich, which company operates a plant at Mombasa with an annual capacity of 400,000 tons and exports to Tanganyika and to various destinations in the Indian Ocean. Through the British Standard Portland Cement Co., Ltd., participation in the construction of a 130,000-ton works near Dar-es-Salaam, which is expected to go into production next year, is being effected. In spite of the difficult conditions in Southern Rhodesia, the Salisbury Portland Cement Co., Ltd., produced better results. In Nigeria, the second kiln of the West African Portland Cement Co., Ltd., which will raise output to 450,000 tons a year, came into production recently. The demand for cement in that territory continues to increase.

A New Cement Works in Northern Ireland.

It is intended that a new cement works is to be established in Northern Ireland by The Associated Portland Cement Manufacturers Ltd. (This development is referred to briefly in the foregoing report of the Chairman's statement for the Annual General Meeting.) The works, which is subject to a development grant from the Government of Northern Ireland, will be situated south of Lough Neagh and it is hoped that it will be operational within eighteen months to two years. It will cost between $\pounds_{3,000,000}$ and $\pounds_{4,000,000}$ and will employ more than twohundred workers.

Information on Cement and Other Building Materials.

THE Blue Circle Group of Companies issued recently a "Data Sheet Manual" which is a complete documentation of information on the thirty-five cements and other building materials made by member-firms of the Group. The materials are divided into the following seven types: Cements and limes; Aggregates and fillers; Admixtures; Adhesives and mastics; Bricks; Plastering products; Decorating products. For each material the information is given in a form to make reference easy, and includes the following:

A brief statement defining the requirements which cause the material to be needed in the building industry; a statement describing the nature of the material (or the materials from which it is made) and the relevant Standards to which it conforms; information showing how the material meets the requirements; available types of each material; examples of where the material may be used; guidance on specifying the material (full specifications are available on application); the treatment and use of the material on site; facilities for supply; and price.

Copies of the Manual are obtainable free from The Cement Marketing Co., Ltd., Portland House, Stag Place, London, S.W.I.

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Accidents in the Cement Industry.

THE Report for the year 1963 by the accident prevention Advisory Committee of the Cement Makers' Federation was issued recently.

The Report states that record achievements are not easy to maintain, and it would be surprising if there were no set-backs in the results achieved year after year by the cement industry in its efforts to reduce the number of accidents. Such a set-back occurred in 1963, the following are the statistics for 1963, the figures in brackets being those for 1962.

Total number of lost-time accidents, 183 (161); frequency rate, 0.69 (0.59); number of works with no lost-time accidents, 4 (12); number of works with a frequency rate below unity, 32 (31). There were only two fatal accidents during the year.

The set-back was not large enough to be significant, the accident total and the overall frequency rate for 1963 being the second lowest on record. It may in part be explained by the abnormal weather conditions in the early part of 1963; out of forty-eight accidents in January and February, eleven were due to, or directly connected with, weather conditions and five were indirectly connected therewith.

Of the total of 183 accidents, only fifty-five occurred in the Blue Circle Group's twenty-seven works, which had an overall frequency rate as low as 0.33. The remaining works had 128 accidents and a frequency rate of 1.29 against the 1962 figures of 130 accidents with frequency rate 1.34.

Only four works succeeded in having a year free from lost-time accidents but nine others had only one accident each. Johnson's and Wouldham works had no accidents thus repeating their record for 1962; Cliffe works has had a clear record for three years. Rodmell works has had two consecutive years without an accident.

An Accident in a Cement Silo.

In the April 1964 number of "Accidents: How they happen and how to prevent them," which is published quarterly by H.M. Factory Inspectorate of the Ministry of Labour, an account is given of a double fatality in a storage silo at a cement works. The silo was one of a row of silos each being 32 ft. in diameter and



Fig. 1.





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about 50 ft. deep (*Fig.* 1). The ladder in the silo was broken and, in order to repair it, the silo had been emptied of cement so far as possible by means of airsuction conveyors. Not all the cement had been removed, however, and some remained in semi-hardened mounds round each of the five 20-in. by 16-in. openings in the flat bottom. It was decided to clear out the remaining cement so as to provide a working space for the erection of scaffolding inside the silo. The chute at one of the openings was removed to enable workmen to enter the silo from below. Lights, safety belts and ropes were then lowered into the silo from the top. The intention was that the men were to work in relays clearing the cement until the foot of the ladder was reached. This was at a horizontal distance of about 15 ft. from the opening by which entry was made.

First one man went in alone, put on his safety belt, and sat part way up on a pile of cement awaiting the entry of the second man, who went in and was presumed to be putting on his safety belt when cement was seen falling through the opening. From the top of the silo, both men could be seen partially buried in cement; they must have disturbed a pile which flowed and engulfed them. The Report comments that both men were allowed to enter the silo without their movements being kept under constant observation. In such cases it is essential that entry be made from the top and that the safety belts be already attached to the men. The safety ropes should be held by a sufficient number of men who keep watch and, at the first indication of danger, are ready to haul the men in the silo to safety.

The Cement Industry in North America.

U.S.A.—A proposal to establish the first cement works in Nevada was reported recently. It will be at Fernley and will be a dry-process plant having a daily productive capacity of 500 tons.

The final phase of rebuilding the cement works owned by the Riverside Cement Co., at Crestmore, California, is expected to be completed in the autumn of this year. The present annual productive capacity of the works is about 500,000 tons; this will be increased to about 750,000 tons. There are at present thirteen kilns but these are being replaced by two 500-ft. kilns. Computer control is being installed.

A fully automated works to produce white cement was opened recently by the Medusa Portland Cement Co., at York, Pennsylvania. It replaces the original works established in 1907. New plant includes a 450-ft. kiln.

The Dewey Portland Cement Co., has closed its works in Oklahoma, which has been in operation since 1920. The operations previously carried on at the works are being absorbed into the enlarged works at Tulsa.

(The foregoing are based on reports in "Rock Products.")

An unusual feature of the new automated plant installed by the Calaveras Cement Co., at their works at Redding, California, is the fact that the 450-ft. kiln is fired by natural gas which is supplied through a pipe-line.

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Canada.—At the Fort White, Manitoba, works of the Canada Cement Co., Ltd., a new kiln which will increase the productive capacity by 50 per cent. is being installed. The annual capacity will then be about 835,000 tons.

A new works is to be established by Canada Cement Co., Ltd., near Brookfield, Nova Scotia. The annual productive capacity of the works is expected to be upwards of 800,000 tons. The brand name is "Maritime" cement.

The St. Lawrence Cement Co., is proposing to extend its works at Quebec. When completed, probably during 1965, the works will have the annual productive capacity of about 735,000 tons.

British American Construction & Materials Ltd., has announced its proposal to establish a new cement works at Rosser, Winnipeg, Ontario. The works, which is expected to be operating late this year, will have an annual output of more than 170,000 tons.

Alaska.—Alaska Portland Cement Ltd., is to establish a works between Anchorage and Fairbanks. The initial productive capacity will be about 85,000 tons annually, but it is expected to double this capacity later.

Mexico.—Allis-Chalmers International have announced that they have received an order to supply equipment for a new cement works to be constructed in northwestern Mexico. The equipment includes a rotary dry-process kiln, which will be 9 ft. 6 in. in diameter and 325 ft. long, and will have an annual productive capacity of about 300,000 tons. Standard Portland cements and a type of pozzolanic Portland cement will be produced. The works, which will be at Hornillos in the State of Sinaloa on the eastern shores of the Gulf of California, is being established by Cementos Sinaloa S.A., a company recently formed by private Mexican investors and the San Luis Mining Co. An adequate supply of raw materials is available close to the site which is some 40 miles inland near the Fuerte River.

(See also references on page 76 to Canada and Mexico).

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