

Physical and Mineralogical Study of Bricks of Historical Masonry Structures of Bhopal, India.

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ABSTRACT: The present study is based on the physical, mechanical and chemical-mineralogical characterization of more than two and half centuryold brick samples collected from Bhopal City of Central India, using mechanical, Field Emission Scanning Electron Microscope (FESEM)Energydispersive X-ray spectroscopy (EDX), and X-ray powder diffraction (XRD) techniques. In all three types of clay brick samples used in this study, the mineralogical phases existed were quartz, Muscovite and feldspars majorly. These bricks were compared with the sample of present day modern samples.

KEYWORDS: Historical building, xrd, bricks, chemical-mineralogical, XRD, XRF,

I. INTRODUCTION

In this era of advanced technological developments, modern structures' diagnosis can be done accurately using various methods and techniques.But the same cannot be inferred for historical monuments and structures. There is a need to acquire a certain level of knowledge about the material used other than the methodologies for precise intervention on historic structures. In ancient structures, the main construction components were lime mortar and brick which played a major role in maintaining structural integrity. Brick masonry played a significant role in the buildout of influential old masonry structuresalong with stone masonry. The masonry not only provides structural stability but also plays a significant role in subdividing the space, thermal and acoustic insulation, as well as fire and weather protection.On the other sidedeterioration and failure of historical structures took place due to various factors such as lack of structural design codes in those days for durability and strength, environmental factors, lack of preservation measures, lack of willingness, and inadequate

maintenance. This state promotes the necessity to evaluate the properties and performance of brick masonry.Research has been carried out in recent times to understand brick masonry's nature and its component materials in different conditions.In unreinforced brick masonry, bricks playa structural part primarily and hence it is required to characterize the bricks to have similar bricks during restoration works of historical buildings. The ingredients required for the preparation of the bricks were readily available since ancient times. The earliest footprints of fire clay bricks are as old as 3000 B.C, referring to early Indus valley cities [1-3]. The compressive and flexure strength of the bricks helps in assessing the mechanical behaviorof the masonry[4].

This paper focuses on determining the characteristic properties of bricks used in Bhopal's historical structures through a series of experimental tests. This characterization of the brick will help in the selection of the new materials further interventions, restoration, for and conservation of the monuments. The incompatible material used can cause the deterioration of the structure, causing irreversible damages[5]. For this purpose, the samples were collected from Chaman Mahal, Rani Mahal, and SaifiaCollege. The Nondestructive testing (NDT) or Mild Destructive Testing (MDT) on the masonry structure or its elements may help assess the masonry and material condition, but it is not always possible in the historical monuments having importance in cultural heritage. Due to their historical value, they are subjected to restrictions to alter, for the conservation of cultural heritage. Owing to this, limited samples could be collected from the building portions as representative samples. The modern-day bricks were also taken for comparison of experimental data. Allthe tests were carried out in accordance with the Indian standards.



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II. HISTORY OF BUILDINGS

SardarDost Mohammad Khan had set up Islamnagar as his first capital and was the founder of Bhopal state.Islamnagarfort comprises of Gondmahal, Rani mahal, and Chamanmahal. ChamanMahal was built in 1710 A.D., and the place is surrounded by an enclosure wall and has two entrances. A sheesh mahal with twelve doors is situated near the entrance. In addition, arches decorated with floral motifs pillared baradari as well as rooms with an open courtyard. A Hammam is also built to the north side of the mahalwithan additional roomthat served the purpose of a dressing room. Furthermore, the second floor of the palace is flat-roofed which consists of two chhatris on either side. Chaman Mahal is a typical mixture of later Mughal, Rajput and Malwa forms of architecture.

Rani Mahal is also an evident form of art built by DostMohammad Khan in the year 1720 which served as a residence for his queens.The west-facing entrance gate is accompanied by an open courtyard and the arched verandahas. A Baradari to the north side is also constructed and has decorated arches of floral motifs.

Sultan KaikhusrauJahanBegum,built a presidential palace called Qaser-e-Sultani (now Saifia College) in the year 1905.

III. FACTORS AFFECTING BRICK CHARACTERISATION

The historic fabrics of the monuments were constructed of brick and lime mortar. Since the destructive testing of building material in the historic monuments is not always possible Nondestructive testing (NDT) or Mild destructive testing (MDT) remains the option.

- a) Colour: some authors have related the colour of the brick to characterise its compressive strength. The hematite being the main colouring agent and main constituent of the clay plays important role. X-ray diffraction (XRD) analysis has shown that red coloured brick has high hematite content as compared to the yellow or light brownish bricks[6].
- b) Firing temperature : usual firing temperature of the bricks is 800°C -1100°C. However the higher kilning temperature results in increase in the vitrification of the clay particles, reducing porosity. Variation in physiochemical, mineralogical, thermal and mechanical properties is there on the basis of the firing temperature. Higher kilning temperature can be detected by presence of mullite and cristobalite minerals in the XRD analysis of the bricks[6]-[11] Since all the

properties are interrelated to each other the chemical composition of the raw material plays an important role in finalizing the final mixfor the clay bricks.

c) Physical and Mechanical Properties as discussed in later section.

IV. EXPERIMENTAL PROGRAM

For the purpose of comparison and analysis sample collectionand coding was done on the basis of the sample origin. The coding for brick samples taken from Chamanmahal, Rani Mahal, and Saifia College were CM-BR(BS 01), RM-BR (BS 02), SC-BR (BS 03) and MD-BR (BS 04), (modern bricks samples) respectively.

The following experimental programme was followed in two stages. The various constituent material present in brick masonry exhibit different behaviour. Therefore, it is important to deduce the individual properties of brick units.

Stage-I:Physical and Mechanical Characteristics 4.1. Visual inspection:

Before subjecting to any test the bricks were cleaned to remove any dust particle or any loose particle/impurity adhered to it. The bricks were dried in the Oven for 24 hrs at the temperature of $103 + 2^{\circ}$ C. Study of the surface texture, colour, particle shape and size of the brocken brick fabrics were recorded.. Then the bricks were subjected to Dimensional analysis using Digital Vernier, Mass using a digital balance & Density. Then Initial rate of absorption (IRA), Water absorption test(WA), Ultrasonic pulse velocity test (UPV) and Rebound Hammer test (RH)werecarried out. In the next stage test for Mechanical Characteristics: Compressive strength and Flexure strength test were carried out after capping the bricks with gypsum plaster to provide flat and parallel face for testing and generation of uniform stress in the sample. The fractured surfaces were visualy examined. Mineral composition using FESEM -EDX, XRDand XRF were determined using the samples of destructive tests.

4.2. Dimension Mass and Density

The bricks collected from the site were found to be handmade burnt clay bricks of uniform shape and size but with warpage. One of the faces was flat and other was not smooth. The dimensions of the bricks have been determined precisely by averaging the value measured at minimum three sections. The resistance of an object to motion varies with the mass which imparts inertia to the object. More is the inertial mass of the object; it provides more stability to the structure. The density



of a material (expressed as mass per unit volume) which is an important parameter which indicates the degree of compactness of material and weight of brickwork. In addition, it also signifies the sinking behaviour of the material.

4.3. Water Absorption Test

The water absorption capacity of the brick is an important parameter for evaluating the performance of the brick. The high absorption values indicate susceptibility to volume changes which results in cracking of the bricks and structural impairment in buildings. Also, it indicates the higher possibilities of building in dampness of the structure. The absorption capacity of bricks is found using the water absorption test as per IS 3495 (Part2): 1992[12]. The weight of each brick samplewas noted as (W1). These completely dried samples were immersed in water a temperature of 27 \pm 2°C for 24 hours. The samples were removed and wiped out with a damp cloth for any traces of water. The weight of individual brick was noted again (W2).The water absorption can be found using this formula:

 $\left(\frac{W_2}{W_1} - 1\right) * 100$ (1)

4.4. Initial Rate of Water Absorption (IRA)

The initial rate of absorption influences the bond between the brick and the mortar . The IRA value affects the structure in many ways. In case, when IRA is low the brick surface in the vicinity of the mortar results in bricks floating on mortar bed.As per Drysdale et al(1994) [13], IRA values give good bond strength in range of 0.25-1.25kg/m2/min and IRA values higher than 1.5kg/m2/min indicates highly absorptive nature of brick.On the other hand higher IRA values results in rapid drying of the mortar. Ravi Teja[14]indicated there exist a correlation between the mechanical properties of the bricks and IRA . He observed that compressive strength of the bricks is inversely prop to IRA and WA ,and direct with dry density. The initial water relation absorption rate is expressed in Kg/m2/min or g/mm2/min .The quantity of water absorbed in first 60 seconds is measured. The guidelines for finding IRA in both field and laboratory is mentioned in ASTM C67 [15]. More investigations are required the permeability to study of bricks. Notwithstanding, it has been recommended that the speed with which water enters a brick bears some connection to durability. Analyses have given some connection among durability and IRA. The IRA test has been effective for anticipating durability. Walls of low IRA brick can be built to have no

water penetration.For walls of uncored brick and excellent workmanship water penetration resistance is directly related to brick density and inversely related to brick absorptions.[16]

4.5. Ultrasonic pulse velocity (UPV) test

The ultrasonic pulse velocity test were conducted as per IS 13311-1 (1992) .[17]. The bricks weresmoothened by rubbing abrasion stone. The surface finish affects the travel time of the pulse and hence a greasing gel was applied between the brick surface and the transmitter and receiver of the UPV machine. The direct test were done along the **longitudinal axes of the samples. The similar**procedure were adopted by[18][19][20][21].

4.6. Rebound hammer test

The rebound hammer test was conducted on the bricks using IS 13311-2 (1992) [22] code. A seating load was applied to the bricks for creating in situ conditions. 10 rebound hammer readings were recorded at different locations on header and stretcher locations with the hammer in the horizontal position. A similar process has been discussed in the RILEMTC MSD 2[23] for European region. Similar tests have been reported by [19][20][21][24].

4.7. Compressive Strength Test

Mechanical tests (uniaxial compression tests) give data on the conduct of bricks acted upon by mechanical stresses and their ability for opposing such stresses. These tests are useful since, after a specific value of stress, bricks can experience irreversible physical harm, adding to the weakening of the material and thus, leading to instability of the structure. Samaransinghe and Lawrence (1992)[25] have studied the compressive properties of low strength bricks.Their experimental study was based on hand moulded bricks drawn from different kilns with almost uniform size and regular surface finish. They concluded that the sizes of the bricks in the same batch of kiln were not uniform and had varying strength. Cultrone et al[26]examined the technical behaviour of bricks using two tests ultrasound and mechanical testing.Kaushik et al[4]studied the uniaxial compressive stress strain behaviour of solid clay bricks of northan India.Bosnjak-Klecina and Loznacic^[27] conducted experiments on old and new bricks for analysis of the physical and mechanical properties of bricks. They suggested applicability of in-situ testing method for determining mechanical properties of bricks. They also inferred the correlation between the penetration speed and material strength as linear.



Nagarajanet. al. (2014)[28]investigated the mechanical properties of bricks and calculated the Young's modulus, stress-strain values of the bricks load.Brozovsky.J under compressive and Zach.J(2007)[29]used ultrasonic pulse velocity to determine the compressive strength of historic bricks as well as new bricks. The effectiveness of method was more for solid bricks having minimum body defects. They also concluded that there was a relation between compressive strength and rebound number.[30]

The maximum load that a brick can withstand is determined by the compressive strength test as per IS 3495 Part 2[12]. The factors on which compressive strength depends are method of brick manufacturing, degree of firing, and composition of clay[28]. The surface of the bricks were made smooth by filling the irregularities of the surface by gypsm capping. The test was conducted by placing the faces of the brick between two ply plywood sheets each of 3mm thickness. The bricks were centred carefully between the plates of the digital compression testing machine . The load was axially applied at a uniform rate of 14 N/mm² per minute till the failure occurs. The maximum compressive strength of each brick is estimated by the load at which the brick fails. The values are noted in (N). The formula for the determining compressive strength is :

Compressive strength = $\frac{\text{Maxi Load at failure}(2)}{(\text{N/mm}^2)}$ Average bed area

4.8. Flexure Strength Test

The flexure strength can be determined experimentally. The flexure strength of three individual brick samples with breadth (B) and depth (D) is evaluated using the formula as per [15][31].

Flexure strength $=\frac{3PL}{BD^2}$ (3)

where L= span between the supports, P = breaking load.

Stage-II: Mineral Characteristics

4.9. Material Characterization Field Emission Scanning Electron Microscope (FESEM)

The morphology of all the samples were observed in model NOVA NANOSEM-430 of COMFEI. For FESEM micrograph samples were fixed on aluminum stubs with carbon tabs. Subsequently, the samples were coated with a thin layer of gold before the observation.

4.10. Energy-dispersive X-ray spectroscopy (EDXA)

The image and the multi elemental content of all the samples were obtained with Model X-MAX of Oxford.

4.11. X-ray Diffraction (XRD)

The small sample of the bricks were crushed to make its fine powder. This powder was placed in RigakuMiniflex II Desktop X-rav diffractometer machine for testing. The testing is done with scanning range of 10 ° to 90 °, scanning rate of $1^{\circ} 2\theta$ /sec.the powder XRD analysis helps in obtaining the brick mineralogy. This technique was initially used by Cultrone et al [18]for mineralogical and physiological characterization of clay bricks with additives. Lopez-Arce et al [32]used the physicochemical characterization for producing new specific brick suitable for restoration using local material in Toledo city. Bhatnagar and Singh [33]evaluated the effect of environmental effects on brick samples using this obtained from the ancient altar in technique Himalayan region. Rai and Dhanpal[34] used XRD with other techniques to find suitability of the renovation at historic monuments at Lucknow. Ahmad et al [10] studied the effect of using local brick raw material for bricks by comparing properties of clays as well as bricks made from them.

V. RESULTS AND DISCUSSION 5.1. Dimension, Mass and Density

The visual inspection of the fracture revealed that the raw material of the bricks consisted of small particles of stones and voids making them hetrogeneious material. The brick sample of Rani mahal was marked with 3 different coloured layer material indicating the non uniform firing of the bricks. After evaluating the bricks of historical buildings it can be clearly seen that all the bricks were by and large of same dimension range but failed in uniformity. The mean values of the dimensions of bricks are shown in [





Figure 1 Dimension (in mm) of Bricks Figure 2 Mass comparision (in Kg) of Bricks Figure 3 Avg densities of brick samples

5.2. Water Absorption

The water absorption values for all brick samples were found within 20% .Thecomparison of water absorption of bricks is represented graphically in the [Figure 4]. The mean value of water absorption calculated from the samples of various historical buildings is found to be 5%. The water absorption value of CMBR is 5.46% which is the highest valueas compared to RMBR and SCBR. The maximum deviation from the mean valueis 11.54% in case of RMBR whereas the mean deviation among samples of CMBR and SCBR is 9.27% and 2.27% respectively.

5.3. Initial Rate of Absorption

The comparative study of initial rate of absorption shows that SCBR has the highest value and RMBR with lowest value. The test setup and samples after test are shown in [Figure 5&Figure 6]. The mean value of IRA from three places is $2.58 \text{ gm/m}^2/\text{min}$. The highest deviation from the mean value is 29.82% in case of SCBR and the deviation among samples from CMBR and RMBR were 11.87% and 17.95% respectively.[Figure 7]

5.4. Ultrasonic pulse velocity (UPV) test and Rebound hammer test

The average values of density, percentage of water absorption, IRA values, RH numbers, UPV and compression strength for the brick samples



aresummarised in[Table 1]. The average of RH indexes for the samples rangedfrom 18-25, while average value of Ultrasonic Pulse Velocities ranges

from 1956 to 2734 m/s.the micro cracks present in the bricks result in the low velocities through them.



Figure 6 Brick samples after IRA test Figure 7 IRA of Bricks Samples

5.5. Compressive Strength

The calculated mean value of compressive strength of brick samples from three historical buildings is 5.95 N/mm². The compressive strength of brick samples from SC-BR is found to be 7.20 N/mm² which is the highest value in old bricks and the percentage variation from the mean value in case of RMBR and CMBR is 12.44% and 5.87% respectively.[Figure 8&Figure 9].The regression analysis was done to obtain a relationship between compressive strength and rebound hammer index. The linear regression equation showed a positive correlation of the two quantities but with the R^2 value of 0.329 depicting not so strong relation. Similarly the regression analysis of compressive strength and ultrasonic pulse velocity also shown a weak relation with R^2 value of 0.44. This is not in

full accordance with as reported by some researchers[30]. [Figure 11& Figure 12]

5.6. Flexure Strength

All the bricks are very poor in flexural strength. The observed mean value of flexure strength of three brick samples from historical buildings is 0.63N/mm². The highest value of flexural strength of brick samples are from SCBR and calculated as 0.69 N/mm².The percentage deviation between the mean value and the highest value is 11.59 %. The flexure strength of brick specimen from RMBR and CMBR are 0.65 N/mm² and 0.56 N/mm² respectively.[Figure 10]. This is attributed to the micro cracks present in the brick structures,



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Figure 8 Comp. strength test setup Figure 9 Compressive strengthof Bricks Samples Figure 10 Flexure strength of Bricks



Figure 11Relationship between comp.strengthand RN of bricks Figure 12Relationship between comp strength and UPV of bricks

Table 1 Test results									
Brick Gr	Place , Year	Colour	Density	WA %	IRA%	σ _b (MPa)	σ _{bt} (MPa)	RH	UPV(m/s)
CM-BR	Chaman	Reddish							
(BS 01)	Mahal	brown	1.35	5.46	3.53	5.21	0.65	21.2	2366.5
	1710								
RM-BR	Rani	Dark Red							
(BS 02)	Mahal		1.18	4.42	2.08	5.44	0.56	19.9	2734.8
	1720								
SC-BR	Saifia	Yellowish							
(BS 03)	College	red	1.23	5.11	3.22	7.2	0.69	20.0	1956.4
	1905								
Average	Historical	-							
values	bricks		1.25	5.00	2.58	5.95	0.63	20.4	2352.6
of									
MD-BR	Modern	-							
(BS 04)	Bricks		1.69	14.54	5.02	9.82	1.0	26.2	3434.7
	2018								

WA percentage of water absorption, IRA initial rate if water absorption, σ_b compressive strength, σ_{bt} average flexure strength, RH average rebound number, UPV(m/s) average direct ultrasonic pulse velocity Figure 10



5.7. Field Emission Scanning Electron Microphotographs-EDXAnalysis-

The scanning electron microphotographs exhibiting microstructure of all the types of samples are studied and are depicted in the Figure 13. All the particles were studied from lower magnification 2x, 5x to higher magnification up to 20 k respectively. The particles are of varying shapes and size there by confirming the presence of multi elemental, multi composition in the sample.[Figure 13]. Mineral phases of the quartz and muscovite were found largely in the micro images.

The EDX of all the samples were studied for their chemical composition at different point in EDX graph and shown as in Figure 13. Furthermore, the EDX pattern of the historic brick sample were found to be carbon, oxygen, magnesium, aluminum, silicon, potassium, calcium and iron with their composition (weight %) 11.95 %, 46.84% , 1.46%, 2.16%, 7.01%, 0.36%, 24.17% and 2.18 % respectively and the elements which are present in the modern brick sample involve carbon, oxygen, magnesium, aluminum, silicon, potassium, calcium ,titanium and iron with their composition (weight %) 11.04 %, 50.63% 0.60%, 3.75%, 10.24%, 1.02%, 20.34%, 0.78% and 1.60 % respectively. Thus, the EDX spectra revealed the predominance of multi component, multi elements like carbon, oxygen, magnesium, aluminum, silicon, potassium, calcium ,iron and titanium in all the samples



Figure 13 EDX- SEMof Bricks -a) Historical building Bricks b) Modern Bricks

The elemental compositions and the microstructural characteristics of the bricks, determined by SEM-EDX analysis are given in [Table 2]. The chemical composition determined by XRFAnalysis shows that the bricks contain

highamounts of SiO2, Al2O3, and Fe2O3, and low amounts of P2O5, TiO2, K2O, MgO, and CaO. [

Table 3]The absence of Ca in their composition supports the notion of the use of calcium poor clays from locally available raw materials sources.



Table 2 Elemental quantification data in bricks samples											
element	С	Si	0	Fe	Al	Mg	KF	W	Ti	NaF	Cu
CM-BR (BS 01)	13.11	31.85	54.23	0.46	0.33	-	-	-	-	-	-
RM -B BS (02)	15.24	20.63	53.58	3.27	5.02	0.78	0.87	-	0.33	0.29	-
SC-BR BS (03)	9.08	17.73	49.2	9.73	8.01	3.19	1.51	0.29	0.68	-	0.59
MD-BR BS (04)	16.31	14.66	49.51	8.3	7.82	1.35	0.98	0.28	0.43	0.37	-
<i>C: Calcite, Q.</i> Albite	· Quartz,	O: oxide	es, Al: Al	uminun	n oxide,	KF:	MAD-10	Feldspar, W	: Woll	Astonite	,NaF:

Table 3 XRF results-Major Content (%) in bricks samples											
Compound sample no	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃		
CM-BR (BS 01)	1.92	16.46	63.16	1.00	2.52	2.58	1.35	0.14	9.79		
RM -BS BS (02)	1.68	15.46	65.16	1.10	2.40	2.47	1.36	0.13	9.50		
SC-BR BS (03)	2.00	16.89	62.26	0.98	2.54	1.98	1.32	0.15	8.53		
MD-BR BS (04)	2.05	17.73	63.05	0.92	2.62	2.09	1.37	0.10	10.61		

5.8. XRD analysis-

XRD technique is one of the mostly used methods to identify different minerals and compounds present in fired clay bricks. Qualitative identification of the phases present in the clay bricks by XRD patterns is relatively easy, although several factors makes complication for accurate identification of the phases present in them. Phases developed during the firing step of the clay bodies are generally correlated with their physicochemical and mechanical properties. On the other hand, mineralogy study of the clay bricks is one of the widely accepted tools for the approximate determination of firing temperatures. In this context, present work was focused to identify the phase compositions of three collected clay bricks from Bhopal city, India using XRD patterns and the results are shown in [Figure 14& Figure 15]. Crystalline phases present in the analyzed three fired clay brick samples were identified mainly of quartz, K-feldspars, hematite and muskovite with the help of the corresponding JCPDS (Joint

Committee on Powder Diffraction Standards) database files[35], [36]. It was observed that after firing at above 900 ° C, a spinel-type phase emerges in clay bodies and the spinel phase gets lost and mullite crystals begin to grow at about 1050 ° C and the spinel-type phase disappears completely with increasing firing to 1100 ° C and the mullite phase becomes quite pronounced. However, the mullite phase is not observed in any of the present samples of the historic buildings. This result indicates that the firing temperature of these bricks should be same. Consequently, it can be said that the absence of the mullite phase in all three fired clay bricks analyzed in this study were fired at low temperature of 850°C and not exceeding 900°C. Calcium compounds were not observed in the XRD spectrum of the sample in line with FESEM- EDX resuls. These results show that bricks were produced by earthen materials containing calcium poor clays and low amounts of calcite minerals.



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Figure 14XRD of Historic Bricks Sample

VI. **CONCLUSION**

Brick is the oldest manmade building material in the world. It is produced by mixing raw materials containing quartz, feldspar and clay with water and then forming, drying and baking. In conclusion, it can be inferred that the absence of standard manufacturing proces and relevant codes are responsible for non uniform size of bricks at the site of investigation. Although, the physical and mechanical properties of historical bricks and present day bricks are not similar and when the brick samples are compared individually there are some inevitable differences. The analysis in this study showed that the bricks present in the historical buildings of Bhopal have a low density (\sim 1.25 g/cm3), a low water absorption (\sim 5%) and low compressive strength values (~6.0 MPa) and flexural strength (~0.63MPa). XRD data show that the bricks fundamentally consists of quartz & muscovite and lesser amounts of hematite &

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Figure 15 XRD of Modern Bricks Sample

potassium feldspar in line with FESEM-EDX analysis. But they definitely differ with the modernday bricks in the physio-mechanical properties which may be resilt of more controlled baking process and standards available.

The research work carried out on historical bricks will provide data for future investigation and selection of bricks in restoration works. Further, studies are needed to be done for practical use of data in retrofitting measures of historical buildings.

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