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Determination of Moisture in Lac by Infra-Red Heating

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DETERMINATION OF MOISTURE IN LAC BY INFRA-RED HEATING

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ABSTRACT. A rapid method for the determination of moisture in lac has been found by using infra-red heating and desiccation under vacuum. A simple apparatus has been devised to effect simultaneous heating and desiccation, so that moisture can be determined in about 1½ hours' time only. The results obtained by this method are the same as those obtained by the time consuming methods at present in use for lac. The apparatus is simple and can be easily assembled in any laboratory with an ordinary vacuum desiccator.

INTRODUCTION

The moisture content of lac is usually small varying from about 1% to 3% depending mostly on the humidity of the surrounding atomsphere and to a lesser extent on some other factors, such as, exposed surface, organic impurities, etc. Although this figure appears to be small, the great influence of moisture on some important physical properties of lac, such as fluidity (Townend and Clayton, 1936) and solubility (Palit, 1940) as well as some chemical properties, such as polymetisability, makes its accurate determination often necessary. It is known, however, that the usual method of drying a substance to constant weight at 100°C or above is not applicable to lac, since it easily gives off its combined water at such high temperatures resulting in a partially polymerised product. Various methods (Rangaswami and Sen, 1942) are therefore in use employing lower temperatures and vacuum for the purpose of determining moisture in lac. These methods, however, are all time consuming and so a rapid method would be definitely helpful to the trade.

With this end in view the dielectric constant method of determining moisture was given a trial some time ago (Bhattacharya, 1947). But it was found that there is no direct correlation between the moisture content of lac and its dielectric constant ; the increase in capacitance due to absorption of moisture being anomalous at a certain stage. It appeared, therefore, that the heating of the sample followed by desiccation was almost indispensable, since usual chemical methods, whereby moisture in solids can be determined, cannot be used for ordinary lac (Gidvani and Kamath, 1945). The various special features of infra-red heating and its successful application to many fields where quick and uniform heating is essential, at once suggested itself for a trial to minimise time in the determination of moisture in lac also.

PRELIMINARY EXPERIMENTS

A large number of experiments were first conducted with samples of lac containing various percentages of moisture to see if infra-red heating could be successfully used to reduce the drying time of lac. A Modinstal* dull emitting infra-red heater, fitted with a reflector was employed for this purpose and it was found that by this method the total time required for the determination of moisture in lac could be reduced from about 24 hours required by the British Standard (B. S. I., 1941) or the Indian Lac Research Institute method (Rangaswami and Sen, 1942) to less than 4 hours only. This period includes infra-red heating of the sample at about 45°C (*i*) either for only 30-45 minutes with regular raking to turn up fresh layers at intervals of 10 minutes, (*ii*) or for 75-90 minutes without any raking of the powdered sample whatsoever, followed by desiccation for 2-3 hours under vacuum at room temperature. The actual values obtained closely agree in all cases with those obtained by the present long-drawn Indian Lac Research Institute method. Further experiments to shorten the period of heating by raising the tempurature appreciably above 45°C were unsuccessful since lac showed a tendency towards softening and consequent blocking. Experiments carried out to dispense with desiccation after heating were also not successful.

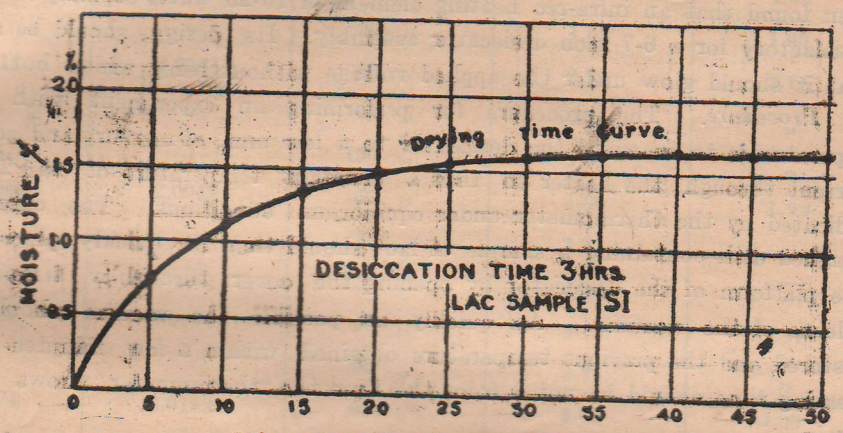
The arrangement was to heat a sample of lac contained in a flat-bottomed dish by means of infra-red rays inside an enclosure such as an ordinary laboratory oven fitted at the top with an infra-red heater and a reflector. A vessel containing fused calcium chloride was kept inside the enclosure to obtain a somewhat uniform ambient atmosphere irrespective of the widely varying humidity conditions that might be present outside during the different seasons of the year. Without a dehydrant in the enclosure the time of heating required to obtain the same result varied from season to season. Immediately after heating the smple was transferred to a vacuum desiccator and carefully weighed at the end of 2 to 3 hours' desiccation. The results were very satisfactory as may be seen from Table I :

TABLE I

Lac Sample	Wt. of lac taken (gms.)	Heating time (mins.)	Desiccation time (hours)	Moisture %	Moisture by Inst. method %	Remarks
S I	2.0352	35	3	1.62	1.60	Raking of the sample at intervals of 10 mins. to turn up fresh layers.
S II	2.0632	30	3	2.21	2.24	
S III	2.0859	35	3	1.90	1.90	
S V	2.0272	45	3	0.97	0.97	
S VI	2.0532	90	2½	2.10	2.05	No raking.

* Modinstal Electric Co., Ltd., England.

It may be noticed that the heating period was about 90 minutes when there was no raking of the sample. With regular raking, however, this heating time might be as short as 30 minutes only. The drying time curve of a sample of lac (S I) has been shown in Fig. I, where it may be noticed that the determined moisture becomes more or less constant after a heating period of 30-35 minutes.



Infra red heating time in minutes

FIG. I

FINAL EXPERIMENTS AND PROPOSED METHOD

Apparatus. It was subsequently thought if it was possible to combine both the heating and the desiccation processes together to save time still further. A special apparatus was, therefore, constructed with a vacuum desiccator as shown in Fig. 2.

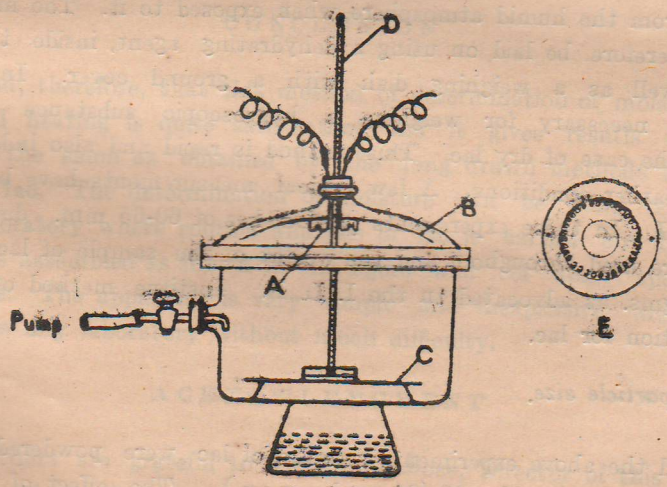


FIG. 2

A is a small ring type infra-red heater made an asbestos-cement circular plate grooved to contain the heater element (shown separately. at E).

A highly polished metal reflector B is fitted at the throw parallel heat rays down on the desiccator platform C below. A thermometer D passes through the rubber cork in such a way that its bulb is only a few mm. above the platform and the dish containing sample of lac can be just placed below without touching it. The pump may be connected with the side tube for evacuation. The desiccant is concentrated sulphuric acid. It has been found that an infra-red heating element of 15-25 watts capacity is quite satisfactory for a 6-7 inch desiccator assembly. Its design should be such that it should glow under the applied voltage without being white hot.

Procedure. The procedure for performing an experiment with this apparatus is to evacuate the desiccator to a few mm. of mercury and adjust current through the heater so that a constant temperature of 45 ± 2 C is indicated by the thermometer under equilibrium conditions. The carefully weighed dish containing a sample of lac should then be quickly placed on the platform of the desiccator by opening the cover through a temporary release of the vacuum. As quickly as possible the vacuum should be restored and the previous temperature obtained within a few minutes. The heating time should be noted from the time the thermometer shows 45°C again.

It has been observed that for most of the samples of lac, one hour's heating under these conditions is sufficient for the purpose of drying. But to be on the safe side 1½ hours' heating will always be found more than enough for complete drying of all samples This means that heating for some extra time when the sample is already dry does not interfere with the determination of moisture in any way. In other words, lac can be dried to constant weight under these conditions. In this connection, it must be clearly stated that dry lac is a hygroscopic substance and it readily absorbs moisture from the humid atmosphere when exposed to it. Too much stress cannot, therefore, be laid on using a dehydrating agent, inside the balance case as well as a weighing dish with a ground cover. In short, all precaution necessary for weighing a hygroscopic substance should be taken in the case of dry lac. This method is rapid and also independent of outside weather conditions. A few typical measurements have been shown in Table II. In these experiments petri dishes of 60-65 mm. diameter with covers were used throughout and the weight of the sample of lac taken was nearly 2 gms. as advocated tn the I. L. R. Institue method of moisture determination for lac.

Effect of particle size.

In all the above experiments samples of lac were powdered to pass a U. S. Standard sieve No. 60 (24 mesh per cm.). The effect of the size of lac particle on the drying time was also studied. It was found that practically no appreciable difference in drying time could be recorded when

TABLE II

Lac sample.	Wt. of lac taken (gms.)	Heating time	Moisture %	Moisture by the Inst. method
S I	2.0272	60 mins.	1.62	1.60
	,,	90 ,,	1.62	1.60
S II	2.0354	60 ,,	2.22	2.24
,,	,,	90 ,,	2.24	,,
,,	,,	120 ,,	2.22	,,
S III	2.0042	60 ,,	1.86	1.90
		90 ,,	1.92	,,
		120 ,,	1.92	,,
S IV	2.0464	60 ,,	3.25	3.27
		90 ,,	3.25	,,
S VI	2.0152	60 ,,	2.06	2.05
		90 ,,	2.06	2.05

the powder was of the above size or finer, but when the size was between 40 to 60 mesh per inch (15-24 mesh per cm.) slightly more time (about 15 mins, extra) was needed to obtain constant weight. Even then 90 minutes' heating time was found to be more than enough for these powders also.

CONCLUSION

We find, therefore, that this method of determination of moisture in lac by infra-red heating is quite satisfactory and it gives resutls which are practically the same as obtained by the long drawn methods at present in use for lac. The determination of moisture can be done very rapidly in any laboratory where routine analysis of lac is carried out if ouly an apparatus is assembled as shown in the figure and set apart specially for this purpose. The apparatus is very simple and inexpensive and can be assembled in any laboratory without much difficulty.

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