



RAPID TURNAROUND OF BET NITROGEN ADSORPTION SPECIFIC SURFACE AREA DATA

BACKGROUND

The client was developing a process for the manufacture of an amorphous API and had identified Specific Surface Area (SSA) as a key product performance attribute. Part of their process development approach involved *Design of Experiments* (DOE) programmes which generated large numbers of samples for analysis. This required precise and consistent sample analysis combined with cost effective and time responsive support, which we supplied despite the client being based in the US. The data had to be of suitable quality to support potential regulatory submission in the future.

EXECUTIVE SUMMARY

The client asked us to work with them because turnaround from local companies in the US was taking too long to meet their requirements. A cost-competitive quote was established with the client for turning around BET Specific Surface Area data within five days from sample receipt. An initial method development exercise was completed to transfer the client method to the Agenda1 Tristar apparatus (Micromeritics), which can simultaneously analyse three samples in a single run. An analytical procedure was documented and approved by the client for routine use to ensure consistent analysis of the samples. Logistically, we identified that when materials were shipped to us on a Friday they always reached us by Monday am, and the analysis of up to 24 samples could then be completed and data returned to them by the Friday. Effective communication was imperative for this project, particularly regarding dispatch tracking numbers, acknowledgement of sample receipt and regular email progress updates with the results laid out in a clear and concise format.

In addition to the routine analysis, we were later asked to collate raw data packages and summary reports for the DOE work, and to carry out other analyses (such as Helium Pycnometry to determine true density) as part of the overall support. This work demonstrated our ability to transfer a method cost effectively and to keep competitive internationally both on price and service levels.

OUR APPROACH IN MORE DETAIL

Our support was based around the use of our cGMP-compliant Micromeritics Tristar apparatus and our previous experience in handling sensitive amorphous materials.

Method Transfer

The client supplied samples of known Specific Surface Area determined by BET nitrogen adsorption.

Agenda1 Analytical Services Ltd.

67 Listerhills Science Park, Campus Road, Bradford, West Yorkshire. BD7 1HR.
Phone: +44 (0)1274 326073 Website: www.agenda1.co.uk



We demonstrated that we could effectively handle these humidity-sensitive materials to reproduce the client results. This required the use of glove box arrangements to maintain samples under controlled low relative humidity conditions. In addition a weekly instrument performance protocol was developed alongside the analytical procedure to verify that the instrument was suitable for use prior to committing samples to analysis. To ensure appropriate resource was available a second analyst was also trained against the analytical procedure.

Sample and Analysis Logistics

The following logistical pattern was established between Agenda1 and the client:

Fri	Mon	Tue	Wed	Thu	Fri
Sample dispatch / tracking number sent to Agenda1.	Acknowledge sample receipt. Verify instrument performance. Start overnight degas of Samples 1-6.	Analyse Samples 1-6. Start overnight degas of Samples 7-12.	Analyse Samples 7-12. Start overnight degas of Samples 13-18.	Analyse Samples 13-18. Start overnight degas of Samples 19-24.	Analyse Samples 18-24. Check all data. Send checked results.

Data Packaging

A summary report of all the data was collated for the client along with hard copies of all the data for PDF archiving.

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Registered Office 11 Murray Street, London NW1 9RE