Dissolution Media Degassing Efficiency Investigation with DissoPrep X8
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Introduction
There are many questions around degassing efficiency of the DissoPrep X8 dissolution media delivery station compared to the recommendation of the USP\textsuperscript{1}. USP proposes a reference method\textsuperscript{2} for degassing of the dissolution media, mainly with parameters like “degassing while stirring heated media under vacuum”. The vacuum is not defined, also not the storage condition. Other validated deaeration / degassing techniques are permitted\textsuperscript{3,4,5}.

Experimental
We have investigated in several degassing tests while the parameters temperature and vacuum (absolute pressure defined) are the main variables. Stirring is performed through a magnetic bar in the storage tank which is digitally driven and software monitored in its function. Duration of degassing is dependant of the reproducible filling time of the storage tank and the additional degassing time which can be selected.

Results and Discussion
The results are listed in the table below. There are 7 tests, but the 7\textsuperscript{th} is an arbitrary test out of the routine which proves the common uncertainty of the oxygen measurement. Therefore, the USP describes only the reference method but not the result of the degassing. Some of the persons in the dissolution lab have reported, that the USP method results with approx. 1 to 2 ppm residual oxygen in the media. But this result is measured in the container of degassing and not in the dissolution test vessel. The DissoPrep X8 delivers media with the measured results of degassing directly into the test vessel, preventing re-aeration while handling the media from bulk storage to the test vessel.
Degassing Efficiency Investigation with DissoPrep X8

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Oxymeter from WTW Ox 330
DissoPrep X8 with Firmware 7.108

Method Parameter | Test No. 1 | 2 | 3 | 4 | 5 | 6 | (7)
--- | --- | --- | --- | --- | --- | --- | ---
Temperature °C | 36 | 45,2 | 22,1 | 36,7 | 45,5 | 36,9 | 37,1
Temperature eff. °C/10 | 360 | 452 | 221 | 367 | 455 | 369 | 371
min. Pressure (vacuum) mbar | 450 | 450 | 90 | 90 | 90 | 90 | 90
Volume mL | 1x 5000 | 1x 5000 | 1x 5000 | 1x 5000 | 1x 5000 | 6x 1000 | 6x 1000
add. Degas Time sec | 120 | 120 | 120 | 120 | 240 | 120 | 120
start Oxygen ppm O₂ | 8.54 | 8.47 | 8.36 | 8.24 | 8.55 | 8.1 | 8.55
end Oxygen ppm O₂ | 6.26 | 5.64 | 5.62 | 3.59 | 3.09 | 3.66 | 3.18

DPX8 routine application
(time saving method without add. Degas Time)

YES YES YES

The degassing is mainly effected by temperature or pressure or both in combination. The edge conditions are there
a) boiling hot water or
b) water under absolute vacuum.

In both cases no air can be dissolved in the liquid. Our test no. 2 was performed with relative high temperature of 45,2 °C and poor vacuum of 450 mbar while test no. 3 was performed under low temperature and high vacuum. Both methods resulted in almost the same degassing efficiency of 5,6 ppm oxygen.

The combination of higher temperature and higher vacuum of the tests no. 4 and 5 resulted in reasonable good degassing efficiency of 3.6 and 3.0 ppm oxygen in the test vessel. In routine application of the DissoPrep X8 the best time saving method (like test no. 4, 6, 7) delivers the most effective degassing result of less than 3.7 ppm oxygen.

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**Conclusion**

The USP reference method and other degassing methods, e.g. with helium, do not provide in routine lower degassing results in the dissolution test vessels as the DissoPrep X8 does it\(^6\).

The residual oxygen in the media is

- according the USP method **1-2 ppm in the container of degassing** – not after dosing in the test vessels
- with DissoPrep X8 in routine application methods without additional Degas Time **less than 3,7 ppm after dosing in the test vessels**.

Besides DissoPrep X8 prepares dissolution media in

- an automated and therefore reproducible procedure,
- metering it directly into the dissolution test vessels,
- warming the media and
- provides a protocol documentation of the dispensing results.
References

1 United States Pharmacopeia  www.usp.org

2 One method of deaeration is as follows: Heat the medium, while stirring gently, to about 41°C, immediately filter under vacuum using a filter having a porosity of 0.45 µm or less, with vigorous stirring, and continue stirring under vacuum for about 5 minutes. Other validated deaeration techniques for removal of dissolved gases may be used.

3 Terry W. Moore, <A fast efficient procedure for degassing dissolution medium>, Dissolution Technologies May/1996

4 Jennifer B. Dressman et al, <Dissolved oxygen as a measure of de- and reaeration of aqueous media for dissolution testing>, Dissolution Technologies August/1998

5, 6 Owen S. Degenhardt et al, <Comparison of the effectiveness of various deaeration techniques>, Dissolution Technologies August/2004